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CONTENTS:

Subject *Page*

Editorials:

Coöperation 145
High Temperature Furnaces 146
Industrial Furnace Section 146
Membership Campaign 147

Original Papers and Discussions:

Some Phenomena in Glaze Reduction—Henderson 148
Natural Hydraulic Cements in New York—Jones 160
Tests of Clays and Limes by the Bureau of Standards Plasticimeter—Kirkpatrick and Orange 170
The Use of Furnace Slag as Grog in Architectural Terra Cotta Bodies—Minton 185
The Effect of Electrolytes on Some Properties of Clays—Schurecht ... 201

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JOURNAL
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Vol. 1 **March, 1918** **No. 3**

EDITORIALS.

COÖPERATION.

The need of technical as well as economic coöperation in the Ceramic industry is unquestioned. In the new era upon which we are about to enter, coöperation will be the keynote of success.

More effective coöperation between our technical laboratories and our manufacturers appears to be one of the needs of the industry. This is especially true in the case of the small manufacturing industries which do not have the services of a technically trained staff at their disposal. Through the lack of technical guidance there has been a great amount of wasted time and effort in some instances. Laborious experiments and costly trials leading to no results have and are being constantly made. The lack of knowledge of some fundamental point has very often spelled the difference between success and failure. In the competition which is destined to come after the War it will be necessary to put our best into the industry. The methods of the past will no longer suffice.

In this connection it would appear that there has been a lack of coöperation and exchange of ideas among our technical laboratories. As evidenced by some of our literature of the past, there has been considerable duplication of researches and wasted effort. In several instances two laboratories have conducted parallel researches covering the same ground. This should not be, as our force of technically trained investigators is small and the field

of Ceramic research is large. Through the activities of the Committees on Ceramics of the National Research Council we have made an excellent beginning in the coördination of the war-essential researches. Why not coördinate the peace-essential researches through a central Research Committee as well?

HIGH TEMPERATURE FURNACES.

To our knowledge a satisfactory furnace for the determination of the softening or deformation points of fire clays and other refractories has not been placed upon the market. The gas furnaces do not reach the temperatures necessary to the softening of our most refractory products. The Deville furnace is unsatisfactory for several reasons. The granular carbon resistor furnaces have not come up to expectations. The vacuum furnace is expensive both as to first cost and maintenance. The method of determination of the softening points is being defined by our Committee on Standards. It is to be hoped that a satisfactory furnace for making the determinations will soon be found. *Inventors please take notice.*

INDUSTRIAL FURNACE SECTION.

The United States Fuel Administration has announced the complete organization of the Industrial Furnace Section, Bureau of Conservation, to handle fuel conservation in all furnaces with the exception of those operated for the production of power, heat and light. This includes those plants using fuel for direct heat, of which the clay products industries are the most important branch.

Mr. A. F. Greaves-Walker has been placed in charge of this Section and has under his immediate supervision 13 districts comprised of 31 states, covering the territory in which industrial furnaces are used. Each district has a local head who has in his organization an advisory board and a corps of inspecting engineers.

The newly appointed district chiefs met at the Fuel Administration Building, on July 29th, for a conference with the chief of the section. The district chiefs made reports based on a pre-

liminary survey of each district. Their conservative estimates of probable annual savings were 3,000,000 tons of coal.

The clay products, lime, cement and glass industries will be inspected first. A standard questionnaire is being furnished each plant owner in advance of the inspection, which he will fill out and hold for the inspector. The rating of each plant will be based upon the efficiency with which fuel is used. All wasteful burning processes will be taken into account, but an opportunity for correcting wasteful conditions will be given before inspection. Recommendations will accompany each questionnaire, several items of which will apply to each and every plant, and if followed will improve conditions materially. The inspecting engineer will be in a position to make further recommendations to each owner after inspecting his plant.

By appealing to the patriotism of the furnace owner, at the same time presenting tangible facts which show him a way of saving money for himself and fuel for the nation's use in the prosecution of the war, little trouble is expected in bringing about conservation of fuel through the proper regulation and operation of all industrial furnaces.

MEMBERSHIP CAMPAIGN

Although we are passing through the vacation period during which it is always difficult to secure action—the Committee on Membership reports the enrollment of an encouraging number of new members during the summer months.

An active campaign for members will be carried on during the fall and winter months and the organization of the industrial and professional divisions as provided for in the Rules will be continued. In this connection it is interesting to note that formal action has been taken by the New Jersey Clay Workers Association leading to their affiliation with the American Ceramic Society.

ORIGINAL PAPERS AND DISCUSSIONS.

SOME PHENOMENA IN GLAZE REDUCTION.

By H. B. Henderson, Columbus, O.

In the development or finish of a number of wares, both decorative and utilitarian, reducing kiln atmospheres are important factors. We may refer to the employment of reducing fires in the production of hard porcelains, in the darkening of salt glazes, the change from dark brown to black, of red burning paving blocks and face bricks, and the golden flash on buff burning clay products.

The production of these effects has become a common practice and within reasonable limits, assuming the use of suitable materials, any desired effect may be produced by variations in the kiln atmosphere and of the duration of the treatment.

We were confronted with this question in making laboratory salt glaze tests on samples of clay. Clays were submitted with the request that we determine whether they were suitable for the production of salt glazed ware. Occasionally we secured favorable results and could report the material satisfactory, but attempts to duplicate the results on the same materials were negative from which it was evident that negative results on a laboratory sample did not safely justify an unfavorable report. The difficulty was explained, however, when we observed that satisfactory results were obtained and could be duplicated under exceedingly smoky kiln conditions accompanied by a deposition of carbon. It occurred to us that colloidal carbon deposited on and absorbed by the viscous surface of the piece might play an important role in the resultant color of the glaze. We know that a very light yellow salt glaze has been obtained in a contin-

uous kiln, under strongly oxidizing conditions, while wares from the same clay in a down draft kiln developed a dark brown glaze. At high temperatures, any kiln atmosphere, regardless of the gross oxygen content, has a somewhat reducing effect upon the wares being burned. If the dark brown color developed by the glaze in a down draft kiln is due to reducing conditions and the consequent presence of carbon, the light yellow color developed by the glaze in a continuous kiln, under normal operation, is also due to a reducing effect, the carbon possibly being the essential factor in both instances, though the quantity of carbon is not an important factor except in the development of color.

The above statement represents the theory upon which we have planned and carried out our investigations and the presentation of the subject at this time is merely a statement of progress without any attempt to draw final conclusions.

INVESTIGATION.

Sewer Pipe.—Samples of salt glazed sewer pipe were collected from different factories, and microscopic slides were made from fragments of the glazes. Under the microscope, even at low magnifications, the glazes were found to contain crystalline material and always of the same crystalline form, namely, hexagonal plates. One would not expect any crystalline carbon to be transparent and it does not seem reasonable that the crystals are graphitic although the carbon present may have influenced the crystallization of other constituents and affected the color.

Chippings of the glazes from pieces of the Akron and Junction City sewer pipe were found to contain 0.01 per cent. of carbon.

Samples of the clean glass drops taken from the roof of a kiln of the American Sewer Pipe Company, Uhrichsville, Ohio, were found to contain 0.014, 0.012, 0.016 and 0.017 per cent. of carbon, respectively. Under the microscope individual crystals, or several of them superimposed, could readily be studied by transmitted light.

In Fig. 1 is shown a microphotograph of a thin section made from a dark, mottled and very brilliant glaze occurring on a No. 2

Fig 1

Fig 2

Fig 3

Fig 4

Fig 5

Fig 6

fire clay body from Logan, Ohio—the dark spots being very small hexagonal plate-like crystals. In aggregates the crystals appear to vary from brown to black in color, but when separated, they appear amber colored by transmitted light. No particular form of grouping is noted.

In Fig. 2 is shown a microphotograph of a reddish colored glaze chipped from a sewer pipe made at Junction City, Ohio, from a mixture of shale and fire clay. Several slides were made from this glaze, using fragments of the normal glaze and also fragments from blotches of glaze which had dripped from the kiln crown. The latter fragments show an unusual growth of crystals and in one instance, where the drippings had collected on a socket of the pipe, the appearance was very similar to that of an iron oxide "aventurine" glaze. We made for comparison an "aventurine" glaze (Fig. 3), and in thin sections the crystals of this glaze appeared to be similar in form and color to those in the sewer pipe glaze, with the difference that the crystals in the former were larger, probably thicker and in consequence the color was more intense. In the "aventurine" glaze the crystals are undoubtedly ferric oxide.

In Fig. 4 is shown a microphotograph of a glaze chipped from a fire clay sewer pipe made at Uhrichsville, Ohio. The blackening of sewer pipe by a strong reducing kiln atmosphere is quite a common practice and excessive reduction has a tendency to produce crazing.[1] An examination of samples of sewer pipe, differing in shade in consequence of variations in the kiln atmosphere, shows an increasing number of crystals correlative to the darkening in color. In case of crazing, it is possible that the glaze, being heavily laden with crystals, is weakened probably by a difference in the coefficient of contraction between the crystals and the glaze matrix and, in consequence, fine hair-like cracks may develop when the proportion of crystalline matter becomes excessive.

In Fig. 5 is shown a microphotograph of a fragment taken from the surface of a fire flashed brick. The golden colors of flashed bricks are developed under the same conditions of kiln atmos-

[1] Karzen, *Trans. Am. Ceram. Soc.*, **18**, 434 (1916).

phere as are those of salt glazed sewer pipe. If the crystals present in salt glazes were the result of the formation of sodium minerals, we would not expect to find them present in fire flashed products, in the development of which no salt is used. Samples representing various stages in the development of fire flashed bricks were prepared by scraping the surfaces with a hard steel tool, the dust thus obtained being mounted on slides.

Small particles of the crystallized material from the surfaces of the sewer pipe and flashed brick may be readily picked up and placed under the microscope by means of a needle point magnet. The magnetic properties of the crystals would indicate the presence of iron[1] in considerable quantity but not necessarily as a constituent of magnetite.

In order to illustrate the relative sizes of the crystals as shown in the microphotographs the following table of measurements is given. The measurements represent in each case the average of a large number of determinations.

Sample No. 1.....	0.00517 mm.	Sewer pipe, Logan, O.
" " 2.....	0.00517 "	Sewer pipe, Junction City, O.
" " 3.....	0.01696 "	Aventurine glaze.
" " 4.....	0.0168o "	Sewer pipe, Dennison, Ohio.
" " 5.....	0.00266 "	Flashed brick.

Fig. 6 illustrates the variation in depth of color encountered on the same product differing only in the degree of burning. "A" is a thin weak glaze; "B" is a glaze of good golden color; "C" is dark brown; and "D" is dark brown and crazed.

A determination of the number of crystals per unit area relative to the number in "A" as unity gave the following results:

"A" = 1, "B" = 6, "C" = 25, "D" = 20.

In D we find many imperfectly developed crystals, and perfect forms are seen entangled in a network of skeleton crystals. This condition is usually caused by very rapid crystallization.

Flashed Brick.—It has been generally maintained that the color of fire flashed ware is due to a chemical change

[1] *Trans. Am. Inst. Min. Engrs., Bull.* 126.

in the iron present in the product, but it is a well-known fact that the color can only be produced by exposing the surfaces of the ware to contact with the flame. The face of a brick so exposed will be beautifully flashed while the ends or back not so exposed, yet not in contact with other bricks, and consequently exposed only to the general reducing conditions of the kiln, will not be flashed. It is impractical to set the bricks in checkers because the flame in passing through the checkers will flash the surface under the open checker while the intermediate sections will not be flashed. The face of a brick so exposed is barred with alternate flashed and unflashed streaks corresponding to the checker work. It is evidently necessary that the flame impinge direct on the surface of the ware, but it does not follow that any dust is deposited on the surface. It might be that the local heating effect of the flame temperature develops a lower degree of viscosity, favorable to the growth of crystals, caused to develop in the reducing atmosphere, and thus furnishing nuclei for their continued growth during the subsequent period of oxidation in cooling. But we are of the opinion that the factor inducing crystallization is extraneous.

A flashed brick when reburned in a normal kiln atmosphere largely loses its flashed color and comes back to the natural buff color. It is a surmise whether the color of the crystalline material is eliminated during the oxidation or simply absorbed in the viscous matrix. We can conceive of the elimination of carbon but it would seem that the iron would tend to give a more intense color under the conditions of oxidation and solution following reduction.

Three samples of fire flashed bricks, all made from the same material and the product of one factory, were selected for examination. One had a pinkish color, which is not desired; one a good fire flashed color; and one a yellowish buff color, or simply a very light fire flashed effect. Under normal fire flashing conditions the pink color predominates but it is "burned out" by oxidation before closing the kiln. In addition to the above we had a series drawn from the kiln as the cooling progressed, showing the progression of the colors relative to the cooling.

All of these samples show the same hexagonal plate crystals found in the salt glaze. The tint of the pinkish colored sample is due to a few overgrown crystals of deep red color scattered among the mass of smaller ones of uniform size and light yellow color. The larger the crystals the redder the color, the very large ones approximating the color of the crystals in the "aventurine" glaze. The normal fire flashed sample contained a multitude of light yellow hexagonal plate crystals of fairly uniform size and color. The light fire flashed sample differs from the normal only in that there are fewer crystals. Trials drawn during the cooling process show a progressive increase in the number of crystals.

Refiring under reducing conditions retained or developed the fire flashed color, but a well flashed brick, refired under oxidizing conditions, largely lost its golden flashed color and developed instead a gray-buff color characteristic of a buff burning clay vitrified under oxidizing conditions.

An "old gold" flashed brick shows a mixture of crystals of different sizes and colors, the larger approaching the red and the smaller having the yellow color.

Reducing conditions are necessary to the introduction or development of the mineral constituents of the crystals, but the crystals are almost wholly developed under oxidizing cooling conditions, not that the oxidizing conditions are necessary—perhaps quite the contrary. We know that in flashed brick the high temperature oxidizing atmosphere not only destroys the crystals but eliminates a factor or so changes the mineral constituents from which h crystals form that we do not get a flashed surface. We also know, from practical experience, that a flashed effect on bricks can be developed at relatively low temperatures, but this effect lacks the true flashed color. Whether the color is due to iron or carbon the reducing conditions give a deposit on the surface of the ware—resulting in a dull lusterless flashed effect. At higher temperature there is surface fusion and in this fused mass the actual growth of the crystals takes place as the ware cools.

Briefly, in producing flashed brick we first subject the ware to reducing conditions, but if we continue this to the end, we sim-

ply blacken the surface. Instead, however, after reducing we burn out some of the black coloring element and start surface fusion. This is followed by additional reduction, partial burning out, the development of a greater degree of fusion, and thus alternating to the end. As an alternative, we carry on the oxidizing conditions until the surface fusion is accomplished, then reduce and finally eliminate any excessive reduction effect by oxidation, and finally cool slowly to develop the crystals.

If the color is due to iron, either in the clay itself or collected from the kiln gases, it is difficult to understand how this color can be burned out by subsequent oxidation except by the assumption of transformation and diffusion effects.

It is inconceivable that, in the cooling stage when the color develops, there can be any accumulation of iron from an outside source, but also inconceivable, were such accumulation possible, that it could be dissipated by subsequent reheating under oxidizing conditions. It seems probable that the intense color is an optical effect due to several modifications of crystals, or that the iron, in solution in the crystal, is in a molecular condition, giving a maximum color effect which largely disappears when the crystalline iron compound is transformed or decomposed. That several modifications of iron oxide compounds occur according to the prevailing oxygen pressure in the atmosphere seems to be an established fact. Again, it is possible that absorption by diffusion into the clay mass may occur.

The color system as applied by Sosman and Hostetter[1] indicates that the red and orange crystals contain an excess of ferric oxide while the yellow crystals have an increasing amount of ferrous oxide.

We must conclude, either, that the fire flashed color and the color of the salt glaze is not due to iron alone or, that the peculiar behavior in its development and disappearance above noted is due to physical changes. If the crystals are induced by carbon, their formation would be prevented under oxidizing kiln atmospheres and they would not develop in the subsequent cooling—unless the carbon be restored by an intermediate reducing kiln condition.

[1] *Trans. Am. Inst. Min. Engrs., Bull.* 126.

Summary.—Our preliminary work shows that both the salt glaze and the surfaces of fire flashed bricks contain crystalline matter; that these crystals have a red to yellow color to which the color of the product is due and represent ferric oxide or other modifications of iron oxide. It is likely that the color of the crystals is due both to a variable iron content, derived from the clay or the kiln gases, and modifications in the crystalline structure with variations in the temperature and oxygen pressure of the atmosphere; that the salt glaze contains carbon; that reducing conditions with the development of free carbon are essential for inducing crystallization; that crystallization hence starts in a reducing atmosphere but continues under oxidizing cooling conditions; that re-oxidation largely dissipates the color effect in flashed clay products.

DISCUSSION.

MR. PARMELEE: Is the firing under reducing conditions more favorable to the production of crystals?

MR. LOVEJOY: Mr. Henderson emphasizes the fact that the chippings from the glaze are magnetic, which indicates reducing conditions. He also quoted a statement made, I believe by Sosman, that the magnetic properties may be due to iron compounds other than ferrous oxide, to which the color may be due. Reducing conditions are necessary.

MR. PARMELEE: May not the presence of metallic iron account for the magnetic properties?

MR. LOVEJOY: No, it is an oxide.

MR. BINNS: Is it presumed that the color is due to colloidal carbon or to iron crystals? Both were mentioned.

MR. LOVEJOY: The author states that the color is due to iron and that the part the carbon plays is not understood. The presence of the carbon appears essential, however, not as regards the color, but as a factor in the development of the crystals. The crystals probably owe their color to the presence of iron. When flash brick are re-oxidized, the carbon present is probably elimina-

ted, and, in cooling, there is no development of crystals. If we reburn the brick under reducing conditions, we can restore the original flashed effect. The introduction of the carbon seems to be the essential feature in the development of the crystals—whether it is a chemical, physical, or mineralogical phenomenon, we do not know. In the absence of the carbon the crystals are not developed.

MR. PARMELEE: There is one point that is not at all clear. We have been shown that there is apparently a resemblance between the crystals developed in the "aventurine" glaze and those on sewer pipe; is it true that reducing conditions are necessary for the development of "aventurine" glazes? If "aventurine" glazes are not developed under reducing conditions, the relationship between the two is not quite apparent.

MR. MINTON: I have produced a number of "aventurine" glazes and have never resorted to reducing conditions. I have always considered that oxidizing conditions were preferable. Mr. Lovejoy emphasizes the importance of employing both reducing and oxidizing conditions.

MR. LOVEJOY: Mr. Henderson merely presented the "aventurine" glaze crystal to show its similarity to the salt glaze crystal. He does not state nor believe, so far as I know, that reducing conditions are necessary to the production of "aventurine" glaze crystals. In one salt glaze sample there was a glitter similar to that in an "aventurine" glaze which led to the comparison. He does not discuss the "aventurine" glaze nor claim any relationship between it and the salt glaze crystal other than crystal form and color. When we first began to use natural gas in the Hocking Valley, difficulty was experienced in securing the rich glaze color that had been secured with coal. The desired result was later accomplished without the use of coal by increasing the volume and pressure of the gas and curtailing the supply of air, in other words, filling the kiln full of unburned gases. My attention has recently been called to a practice which has been developed in connection with the burning of fire-flashed brick with coal. At the latter stage of the burn, a quart or more

of heavy oil is introduced into each furnace, the result being a very beautiful golden-flashed brick. The carbon undoubtedly plays some part in the development of these colors.

MR. WASHBURN: Attention has been called to the presence of carbon as a necessary condition in the development of the color. The effect of the carbon is to produce a certain definite concentration of carbon monoxide, and it is in this indirect manner that the reducing action takes place. The carbon itself does not appear to play any direct part in the action, but insures a definite concentration of carbon monoxide which apparently is the active reducing agent.

MR. BINNS: Some of those here can recall, in connection with the work of this Society, about 18 years ago, a discussion in reference to the development of flashed brick, and one of the members displayed a series of trials, drawn at periods from a kiln, showing that the trials when first drawn were blue, but the color gradually changed to yellow in cooling. Founded upon that fact, a theory was advanced which has been more or less the subject of discussion ever since—the formation of ferrous silicates. The thought which has been passing through my mind in that connection, applying to both this case and that of flashed brick, is that ferrous silicate has evidently formed and may be oxidized to the ferric silicate. If this is true, it accounts for the phenomena of flashed brick and also the yellow crystals in a carbonaceous magma. I think that this point explains the difference between the "aventurine" glaze and the crystal in the sewer pipe. In the "aventurine" glaze, the iron is added to the glaze and is readily available; in the sewer pipe the iron has to be dissolved from the clay and can only be dissolved when it is in the ferrous form. I have produced successful salt glazes in an experimental kiln without any reduction and have secured brown colors quite easily with no more reduction than is caused by a partial closing of the damper.

MR. STULL: Certain shades of amber glass are produced by dissolved carbon and the presence of a small per cent. of carbon in the glaze would account for the amber color. We know that

iron, under certain conditions, will also produce a yellow color. It is possible that the yellow color is enhanced by the presence of the dissolved carbon.

MR. BINNS: Why is it then, that in the burning of porcelain, the carbon stains the glaze black? The greatest difficulty in burning porcelain successfully, under reducing conditions, is the elimination of the carbon dissolved in the glaze. Why is it that the carbon stains the sewer pipe glaze yellow, and the porcelain glaze black?

MR. STALEY: In regard to the formation of ferric silicates, I would say that one encounters statements in ceramic literature to the effect that ferric silicates cannot be formed and that ferrous silicates are readily formed. We have, in mineralogical literature, repeated references to ferric silicates. In acid glazes one can introduce red ferric oxide, which, upon heating in the electric furnace, produces a yellow glass. There is no question in that case of the formation of a ferric silicate, without previous formation of ferrous silicate.

MR. BINNS: Mr. Staley has no proof that it is not a ferric solution.

NATURAL HYDRAULIC CEMENTS IN NEW YORK.

By Robert W. Jones, Tompkinsville, N. Y.

It is impossible to determine the exact date when natural rock cements first came into use. In modern times the first structure, of any importance, in which this material was used was the Eddystone Light House, constructed by John Smeaton. The original discovery by Smeaton was in 1756. It was not until 1796, however, that the actual production of natural cements was made on a commercial scale. This material, produced from septaria, was patented by a Mr. Parker, under the name of "Roman" cement. Twenty-two years later the first production was made in the United States. The growth of the industry, in this country, has been closely associated with the construction of canals and other improvements of our inland water ways.

Limestones, after being burned, produce materials which can be conveniently classified under four heads, resulting chiefly from the varying amounts of impurities in the form of silica, iron and alumina. This classification includes common lime produced from the comparatively pure calcium or calcium-magnesium limestones; weak hydraulic limes carrying from ten to twelve per cent. of impurities; strong hydraulic limes with from eighteen to twenty-two per cent. of impurities, and hydraulic cement carrying as high as forty-two per cent. A large production of material reported from New York State as hydraulic cement should have been classified under hydraulic limes. The production of hydraulic limes, as a commercial industry of any magnitude, ceased many years ago. The following figures of production have been corrected, as far as possible, in regard to this production.

In 1768 was the first official notice of the desire for improvement of the inland navigation of the present State of New York. It was not until 1791, however, that any official act was passed looking towards the fulfillment of these desires. In that year there was appropriated by the State Legislature the sum of $250 for an exploration and survey along the line of the present Barge

Canal. This work was completed under the direction of Benjamin Wright. In 1816 Mr. Wright was assigned as engineer in charge of the middle section of the Erie Canal with jurisdiction extending from the Seneca river to Rome, N. Y. Mr. Wright had as assistant engineer Mr. Canvass White. Mr. White, who was an engineer of great industry, made a voyage to Europe, at his own expense, to study canal construction. While in Europe the use of Taris and Roman cements, in hydraulic construction, was strongly brought to his attention. Mr. Benjamin Wright had recommended the importation of these cements for use on the Erie canal, but the great expense attached to this importation had worked against it and attempts were being made to complete the rock work with lime mortar. In most cases this was attended with complete failure.

In 1818 Mason Harris and Thomas Livingston entered into a contract to furnish lime for the middle section of the Erie canal. A quarry was opened on the property of T. Clark, Chittenange, Madison County. The burned material from this quarry lacked the usual characteristics of the ordinary lime in that it did not slake. Samples of this material being brought to the attention of Mr. White were recognized as being similar to the English Roman cements with which he was so familiar. After considerable experimentation patents were issued to Mr. White on February 1, 1820, and February 17, 1821, for the manufacture of natural cements for hydraulic purposes. There was considerable reluctance on the part of the Canal Commissioners and more on the part of the masons towards the use of this hydraulic cement. After a number of failures in the use of lime mortar, however, it came into universal use. In 1825 Mr. White brought suit against several contractors for non-payment of royalty, amounting to $0.04 per bushel, and secured judgment. The contractors petitioned the legislature to relieve them of these judgments. The joint committee on Canals and Internal Improvements, believing in the justice of Mr. White's claim, recommended that he be paid the sum of $10,000 to secure, to the people of the State, the free use of his cement. Mr. White accepted and a bill was introduced on February 11, 1825, read the first and second time, and committed to the committee of the whole

House. The bill died there and no further attempt was made to secure the payment.

In 1823 Mr. Wright was instructed to make a canal survey from tidewater, at the mouth of the Walkill, in Ulster County, to the Delaware river. This was to be known later as the Delaware and Hudson Canal. Construction began in 1825. Chittenango cement was used in the rock work during that year. Later in that year hydraulic cement rock was discovered near High Falls, Ulster County, but it was not until the summer of 1826 that the first commercial kiln was burned. This was at High Falls, and was operated by John Littlejohn. From this came the great industry which made Rosendale cement known, throughout the United States, as the standard natural hydraulic cement. After the completion of the rock work along this canal the local cement industry declined for a number of years and, until 1847, was carried on in a small way by a great number of producers. In that year came the first combination of operators and the production increased rapidly from an annual output of 200,000 barrels until in 1898 there was produced, from this section, almost 4,000,000 barrels.

For many years, after the discovery of the natural cement rock at Chittenango, there was great activity in the natural cement industry. Rock suitable for the production of this material was found at many localities along or in the neighborhood of the Erie canal and kilns were soon in operation throughout nearly the entire length of the canal. Soon after construction was begun on the Champlain canal, cement rock was discovered at Galesville, and quite a production was made for use on this canal. The construction of the Black River canal was responsible for the discovery and production of natural cement at Lowville, Martinsburgh, Depeauville and Waddington. This section supported quite an industry, although it declined rapidly with the close of canal construction. The more prominent localities along the Erie canal were Grand Island, Williamsville, Buffalo, Akron, Lockport, Oak Orchard Creek, Chittenango and the localities in and around Onondaga County. These were Fayetteville, Manlius, Edwards Falls, Jamesville, Marcellus Falls and Skaneateles Falls. Fayetteville began to produce in 1820 and

Williamsville in 1824. In 1839 Jonathan Delano erected works at Falkirk, near Akron, Erie County. In 1843 this business was passed on to James Montgomery and later transferred to Enos Newman. In 1854, H. Cummins and Son erected a plant at Akron. This was succeeded in 1865 by another plant. The Akron Cement Company was organized in 1870, taking over the plants of H. Cummins and Son. A few years later Cummins Brothers erected another plant west of Akron. The production

FIG. 1.—Behan's quarry, Manlius, Onondaga County, New York. The upper level of the quarry floor and the second heavy bed from the top of the quarry face were the productive cement layers.

of cement was carried on at Akron until 1909. This section was probably the greatest producer of hydraulic limes in the State.

The first cement made in Buffalo was produced by Warren Granger, in 1850. Lewis J. Bennett began the production of cement and organized the Buffalo Cement Company in 1874. This was an active producer of cement until 1902. The plants in Erie County are now all abandoned. The only other section, along the Erie canal, to become a permanent producer, was the Onondaga

County section. Production began at these localities almost immediately after the original discovery and has been continuous to the present time. The output of this section falls in a class between the true hydraulic cements and the hydraulic limes. It can hardly be classed as a true cement, although its characteristics place it closer to the cements than the limes.

The only other section making a continuous production was that of Howe's Cave, Schoharie County. The first kiln was

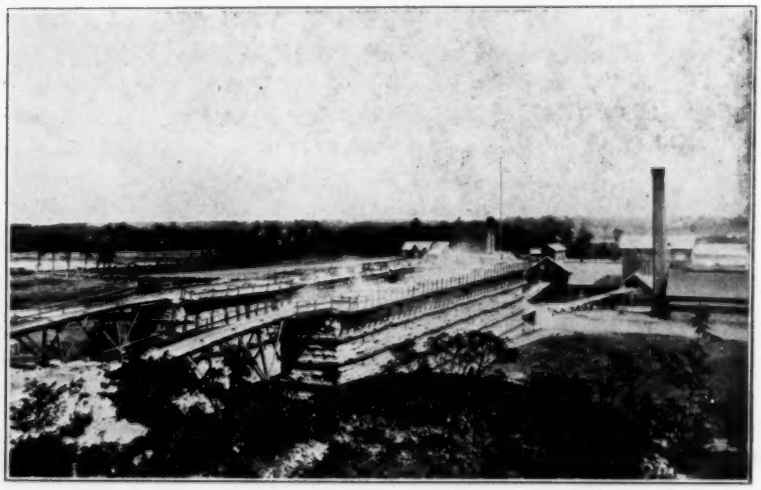


FIG. 2.—A continuous type of cement kiln, Cummings Cement Co., Akron, Erie County, New York. This was the type of kiln used by all of the Erie County producers.

burned at this locality by Mr. Vrooman in 1867. A few years later another plant was placed in operation and the two were productive until 1896, when they were consolidated and, after considerable reconstruction, production was again started in 1898, by the Helderberg Cement Company and carried on until 1905. At the present time, in the State of New York, there is one plant in operation in the Rosendale District and three in Onondaga County. The indications are that only one company

will celebrate the one hundredth anniversary of the discovery of natural cement in this State.

Geology.—The production of natural hydraulic cement has not been limited to any one formation, but includes limestones from the middle Ordovicic to the upper Siluric. The lower, or oldest in the geological scale, is found in the Black River beds. The production from these beds was short lived and was consumed almost entirely in local construction. This material is included among the true cements, although of a rather poor grade. These

FIG. 3.—Opening in the cement layers at Akron, Erie County. A heavy layer of Onondaga limestone forms the roof.

beds have been productive to the extent of approximately 250,000 barrels. Next in ascending order comes the Rosendale water-lime, a subdivision of the Cayugan group of the Siluric. It is from these beds at High Falls that the original discovery, in the Rosendale region, was made and has been responsible for a production greater than any other section. Next in order come the Rondout water-limes of the Cayugan group. Rondout water-limes are exposed in the region between East Kingston and Kingston and have been extensively worked. The production from Buffalo,

Akron and Howe's Cave came from this horizon. The formation highest in the geological scale, which has furnished a commercial natural cement, is that of the Manlius limestone. Near the top of this formation, in Onondaga County, are found two thin beds from which the entire output of this section has been produced.

Mining.—In Onondaga County, due to the few feet of material available, the production of crude rock has been secured from open cuts and presents no features of unusual interest as regards min-

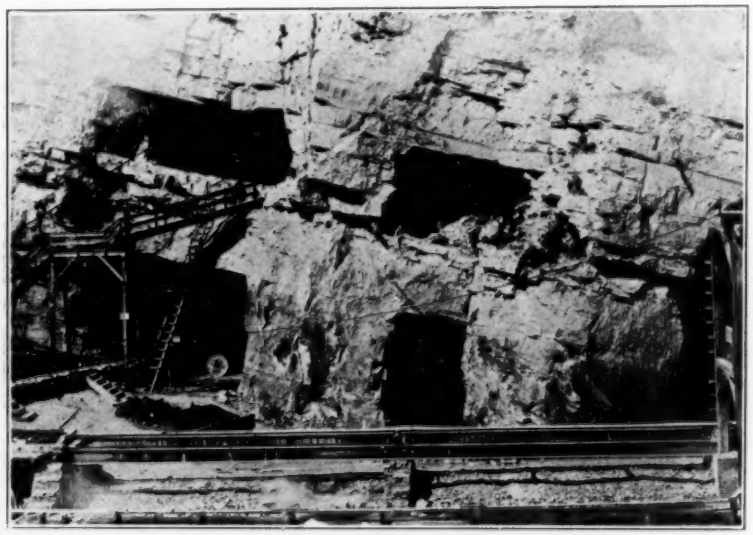


FIG. 4.—Openings of cement mines at Rosendale, showing method of mining along the productive beds.

ing. In the Buffalo-Akron district three of the four producers secured their crude material through underground mining. Adits were driven in along the outcrop, leaving heavy pillars of cement rock for support. The cement rock varied in thickness from five to eight feet. The roof, being of massive limestone, required only a very small amount of timbering. The Howe's Cave material was secured in the early days by open cuts and later by underground mining. It was in the Rosendale-Rondout

region that the greatest difficulties were encountered in the production of crude material. In this region the formations have been folded and faulted to such an extent that the entire output has had to be raised through inclines to the kilns. A distance of over eight hundred feet along the incline has been reached in one instance. The mine of the Consolidated Rosendale Cement Company, at Binnewater, is a typical example of mining in this region. At this property the main incline has an inclination varying from thirty-five to fifty-five degrees. There has been no attempt made to place the different working levels at any regular interval although the distance does approximate one hundred feet. On arriving at a point on the incline where it has been determined to drift along the beds, a cross-cut is driven across the three workable beds, which are known locally as the "old dark," "new dark" and "light." Between the "old dark" and the "new dark" beds is another bed of "light" which, on account of poor mining conditions, is not productive. Drifting is carried along the three beds in a northerly and southerly direction and at approximately right angles with the cross-cut. The slight grade of the drifts towards the cross-cut throws the drift line on a slight curve towards the east in order to keep out of the foot wall. At a distance of about sixty feet north and south from the cross-cut, along the "old dark" bed, inclines are driven downward along the bed. On reaching the determined new level, drifts are run connecting the two inclines and a raise is then driven along the bed in line with the main haulage incline. Rails are laid as the work progresses and, preferably at the end of a Saturday day shift, connection is made. On Monday the new level and incline is ready for use. At each level the opening of the haulage incline is equipped with a hinged door so arranged that when lowered the descending car passes over into the cross-cut and when raised allows the car to pass to the lower levels. There are six levels, the lower being used as a sump, the two upper not being productive on account of poor hanging walls. Very little timber is used and only at points where the hanging wall shows evidences of weakness. The working faces are carried with a width of about twenty-five feet and a height of eighteen feet. The entire output comes from the drift face and no attempt is

made towards stoping. Operations on the first and second levels had, in the early days, been let by contract with very poor results. The first level has been carried to the surface by frequent raises along the bed with very small pillars placed at irregular intervals and leaving very large rooms. It had been the intention of the present Company to mine the lower levels, leaving pillars of not less than forty feet in width.

Burning.—In the early days the raw material was broken roughly by hand and burned in crude upright kilns similar to those used in

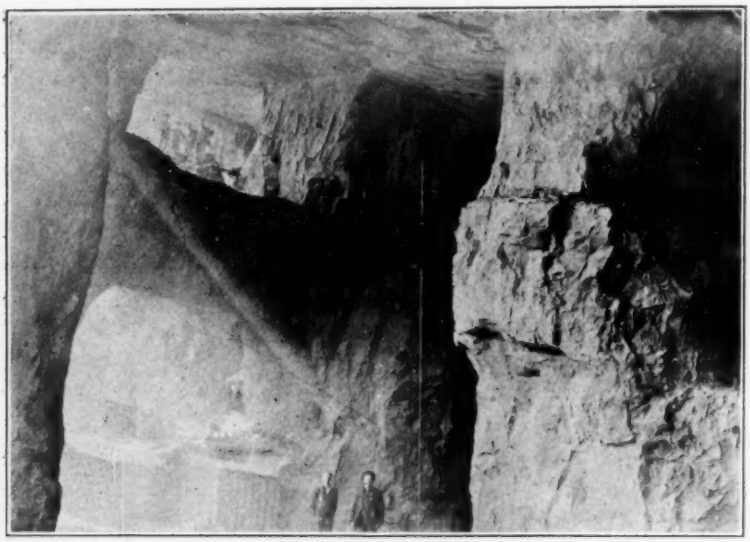


FIG. 5.—Interior view of mine at Rosendale, Ulster County, New York.

burning lime. The walls were usually built of limestone and the kilns were sometimes lined with brick. Wood at first was the only fuel economically available. The fuel consumption was high and as soon as coal became more plentiful it was substituted for the wood. With the use of coal came the permanent upright kiln into which the crude rock and coal were fed in alternate layers and a certain amount of burned material removed every day. Rotary kilns were never used. The burned material was hand-picked, the underburned being fed back to the kilns or to a

special kiln and the burned being passed through jaw crushers and finally to millstones. After ageing, the material was shipped in paper sacks or wooden barrels.

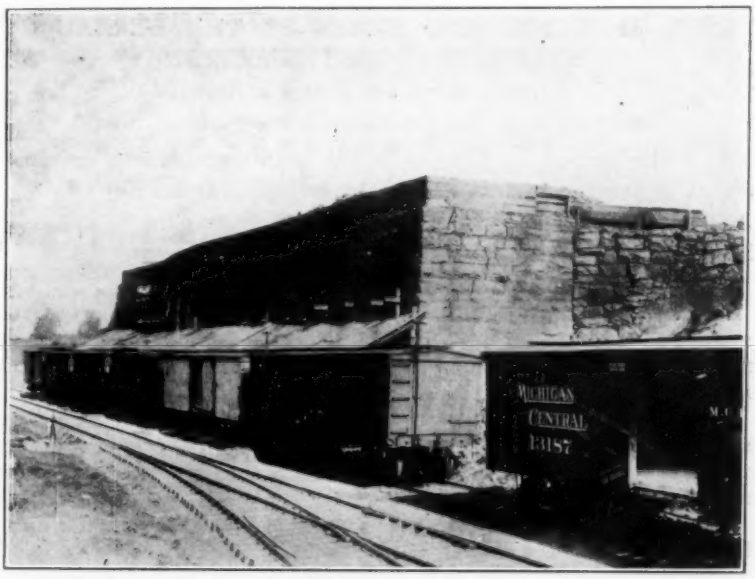


FIG. 6.[1]—Alvord and Company, Manlius, Onondaga County, New York. Old style of intermittent kiln now abandoned except in this district.

The following figures of production have been taken in most cases from original sources and may be accepted as the correct number of barrels of natural hydraulic cement manufactured in the State of New York since the discovery in Madison County. No attempt has been made to calculate the value per barrel. The production by districts is as follows:

Rosendale-Rondout district	85,873,000	Barrels
Erie County	14,300,000	"
Onondaga District	17,000,000	"
Howe's Cave	1,250,000	"
Miscellaneous	900,000	"
Total	119,323,000	"

[1] The preceding illustrations have been taken from cuts which have appeared in various reports of the New York State Museum.

TESTS OF CLAYS AND LIMES BY THE BUREAU OF STANDARDS PLASTICIMETER.[1]

By F. A. Kirkpatrick and W. B. Orange, Pittsburgh, Pa.

[1] By permission of the Director, Bureau of Standards.

It is the purpose of this discussion to give the recent developments in the theory and use of the instrument described by W. E. Emley in the Transactions of this Society,[2] as the "Bureau of Standards Plasticimeter" as follows:

[2] *Trans. Am. Ceram. Soc.*, **19**, 523–33 (1917):

"When spreading a plaster on a wall, the trowel is so held that it makes an angle of 10° to 15° with the plane of the wall. The force applied to the trowel is thereby divided into two components, one acting parallel to the wall—the other acting normal to it.

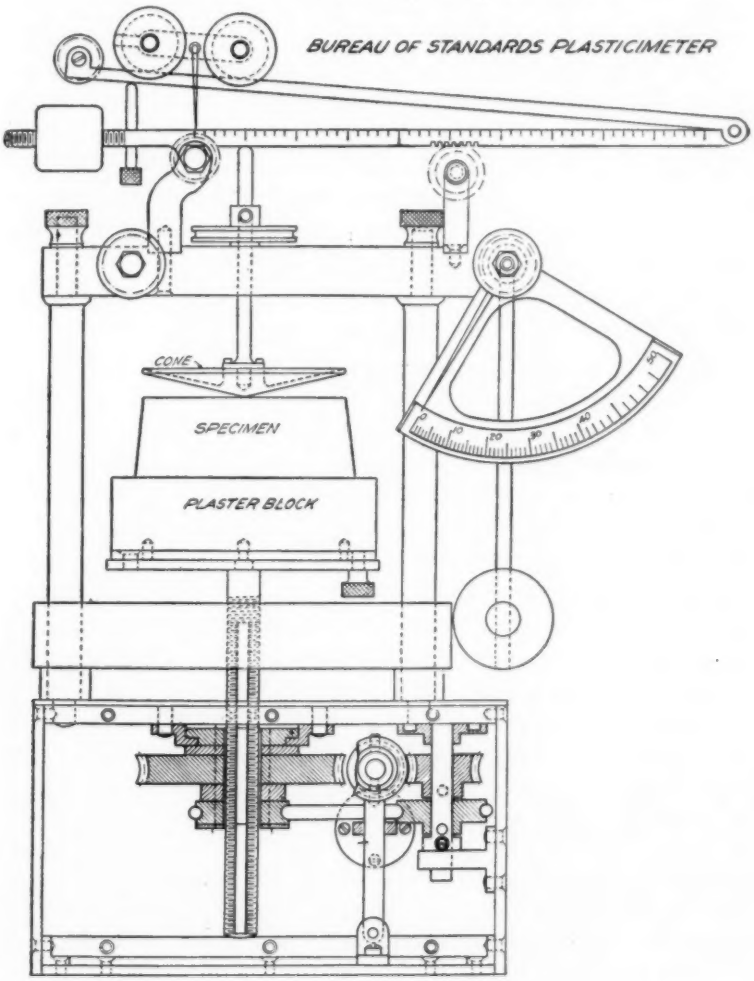
"In the plasticimeter, the trowel is represented by a conical disk, which is mounted point downwards on a vertical shaft. The side of the cone makes an angle of 10° with the horizontal corresponding to the angle between the trowel and the wall.

"The wall is represented by a disk of plaster of Paris of known absorption. It is mounted on the upper end of a vertical shaft, directly below and concentric with the cone. This shaft is threaded and runs through a fixed nut. As the shaft revolves, the wall (or disk) is forced against the trowel (or cone) with a spiral motion. It presses vertically upwards against the cone in a manner analogous to that component of the force exerted by the trowel which acts normal to the wall. The turning motion moves the disk over the face of the cone bringing into play that component of the force which is parallel to the wall.

"The cone is so mounted that either of these forces is able to cause motion independently of the other. Any motion of the cone is transmitted through a system of levers to one of two bars, which are suspended like pendulums, and causes the bar to swing out of its originally vertical position. The greater the angle through which the bar is moved, the greater will be the force of gravity tending to return it and the cone to their original position. It follows, therefore, that if a constant force tends to cause motion of the cone, the bar will swing out until the force of gravity acting on the bar is in equilibrium with the force acting on the cone. When this condition has been attained, both the cone and the bar become stationary, and the force acting on the cone is directly proportional to the sine of the angle through which the bar has moved. The bars are graduated and provided with bobs which can be moved up or down thus decreasing or increasing the force required to produce a given angular deflection.

"Calculating from the dimensions of the instrument as at present designed, we find the following relations:

v = 1.6 + 27.42 sine a + 4.65 1 sine a.
t = 9.29 sine a + 1.577 1 sine a.
v = vertical force acting on cone, in grams per sq. cm.
t = tangential force acting on cone, in grams per sq. cm.
a = angle which the bar makes with the vertical.
l = distance from point of support of bar to top of bob, in cms.



"The independent term in the first equation is caused by the weight of the cone itself.

"If a sample of plaster is molded on the disk, and is driven against the cone at a constant speed, the two bars will assume definite positions. From these positions, the two forces acting on the sample may be calculated by means of the above formula. The vertical force corresponds to that component of the force actuating a trowel, which is normal to the wall, the tangential force corresponds to the component parallel to the wall. The machine is, therefore, capable of measuring independently the different forces which are applied to a trowel."

In the use of lime the word "plasticity" may be used to indicate the intrinsic plasticity of a paste which is not losing water to an absorbent surface; the rate of change of plasticity of a paste spread upon an absorbent surface; or the degree of workability of lime mortars. In each of these three cases different laws apply to the observed behavior of the materials. Hence, until more work has been done with the plasticimeter and the word "plasticity" is defined for the various materials, it is not advisable to offer the instrument as a universal means of measuring plasticity.

In the case of clays, it has not been determined whether or not the plasticity is actually measured by this instrument. The experiments with clays in the plasticimeter have been based upon the changes in plasticity when in contact with an absorbent surface. It was hoped that by the use of this instrument the relative values of clays and pottery bodies in the process of jiggering could be measured.

The authors of the present paper do not agree with Mr. Emley's calibration of the instrument so far as the tangential forces are concerned. In making this calibration he assumed that the forces acting in the horizontal plane, between the disk and the specimen, were of uniform magnitude in each unit area of the disk surface and hence that the total force could be considered as concentrated at a distance from the center equal to the radius of gyration of the surface of the disk. It is well known that the forces of friction vary with the speed. The speed of different portions of the specimen varies as the distance from the center and the forces of friction and adhesion are also subject to variation. The laws of these variations for lime pastes are not known

and considerable work will be required to determine them.

In view of these facts we have not used the equation given in Emley's article: In the present work the distance l was 12 cm. and was not varied. The sine of the angle a was used as "the relative tangential force." This method gave numbers which were proportional to the actual forces involved and eliminated the uncertainty of the calibration originally proposed. A measurement of the vertical forces has been found to serve no useful purpose.

Valuable results have not been obtained for either clays or limes by the use of a non-absorbent surface. In the tests herein reported a plaster of paris absorbent surface was used. As will be shown, contrary to Mr. Emley's contention, the results were found to depend upon the consistency of the clay body or the lime paste.

The method for determining the sand carrying capacity of the lime, as proposed by Mr. Emley, was found to be impracticable. Since the absolute value of the tangential force is not known, the work done upon the specimen cannot be calculated, although the general principle of the relation of work to the plasticity determinations in the plasticimeter still holds.

The method at present employed in the calculation of a plasticity or working quality figure for limes or clays is as follows:

The average relative tangential force for the period of time from zero to five minutes is called the "plasticity figure" of the clay or lime being tested. This method gives the most satisfactory values in a comparison of the plasticities of materials. If the limit of the machine is reached before the end of the five-minute period, the remaining values are obtained by extrapolation.

Method of Testing.

Limes.—Pastes were prepared from each hydrated lime. Each paste was covered with a damp cloth and, after standing at room temperature for from 16 to 18 hours, was molded into test pieces 3.15″ in diameter and 1.5″ high, the base being a plaster of paris block. The test pieces and plaster of paris block were then

placed in the instrument and revolved. As the water was absorbed by the plaster, the viscosity of the paste increased and the forces became greater—at a rate depending upon the rate of absorption. The relative tangential force was noted at intervals of 15 seconds—the instant at which the operator began to mold the specimen being taken as zero time.

In Fig. 1 are graphically shown the results secured on eleven hydrated lime pastes, the time being plotted against the relative

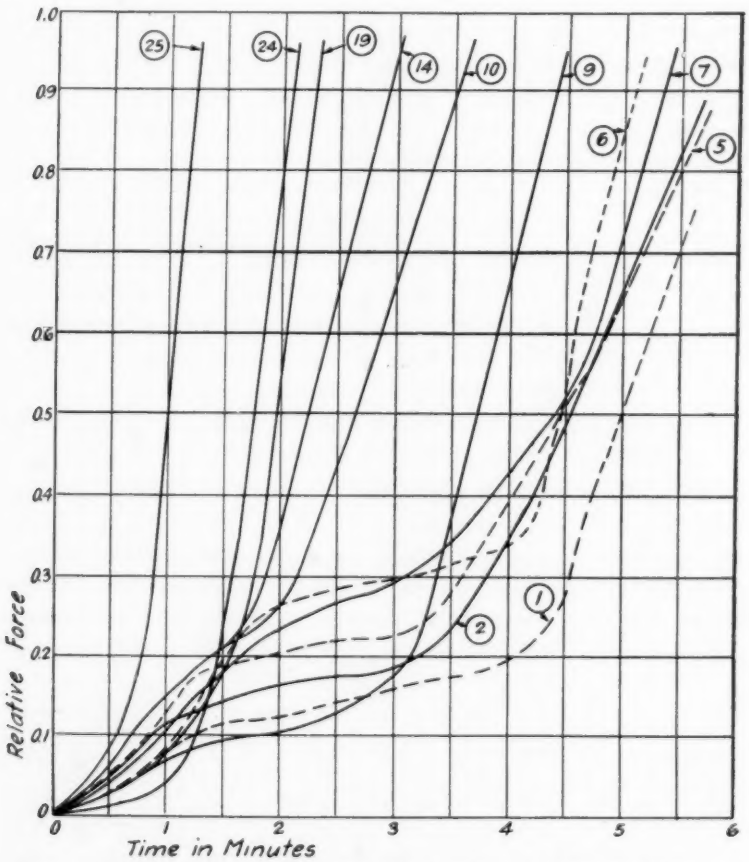


FIG. 1—Relation between time and relative force for hydrated limes in the Bureau of Standards Plastici meter

tangential forces. Since there is no accurate method of determining the degree of consistency of the pastes, three different consistencies were used and the one having the smallest value was chosen for comparative purposes.

Specimens Nos. 1 to 9, inclusive, are representative of plasters capable of being used commercially as finishing coats on walls. Up to a period of from 3 to 4 minutes the forces for these specimens are low after which they increase rapidly. Specimens Nos. 10 to 25 are not representative of commercial finishing coats. The forces for these latter specimens increase rapidly from the start, the form of the curves distinguishing them from those of the finishing limes. The average relative force for the period from zero to five minutes was calculated for each lime and the lower this average force, or "plasticity figure," the greater the plasticity of the lime. The results of the tests on the 25 hydrated limes are presented in Table 1. There seems to be no doubt that the plasticimeter has drawn a distinguishing line between the finishing and the non-finishing varieties of hydrated limes and has determined their relative behavior when spread upon absorbent surfaces.

Lime-Gypsum.—Owing to the fact that, in the preparation of finishing coats, burned gypsum is always added to hydrated lime, it was thought desirable to determine the working properties of lime-gypsum mixtures and the effect of the amount of water used in the preparation of the pastes. An Ohio finishing hydrated lime was mixed with amounts of water varying from 34 per cent. to 56 per cent., by weight. Each paste was allowed to stand from 16 to 18 hours and the "plasticity figure" was then determined in the plasticimeter. To a portion of each paste was then added 25 per cent. of burned gypsum (by weight of the dry hydrated lime), 0.25 per cent. of organic retarder (by weight of the dry burned gypsum), and sufficient water to give all of the pastes a like degree of consistency. This consistency was made to approach, as nearly as possible, that preferred by the plasterer in the coating of walls.

These mixtures were also tested in the plasticimeter. Some of the pastes were too soft to be molded and were placed in the machine in a container with the plaster of paris block at the

bottom. In this series of tests the test specimens were one inch thick instead of the $1^1/_2$ inches as ordinarily used. The results of these tests are given in Table 2 and shown graphically in Fig. 2. The percentage water in the lime-gypsum mixtures is not taken into account in the diagram, as these mixtures were all of practically the same consistency and water content.

TABLE 1.—"PLASTICITY FIGURES" OF HYDRATED LIMES.

No.	Source. (*a*)	Kind.	Plasticity figure.	Per cent. (*b*) water used.
1	Pennsylvania	[1]H M	0.163	42.5
2	Ohio	[2]H M F	0.227	43.6
3	Ohio	H M F	0.241	42.9
4	Ohio	H M F	0.244	43.6
5	Ohio	H M F	0.248	43.4
6	Ohio	H M F	0.277	44.4
7	Ohio	H M F	0.297	42.9
8	Vermont	[3]H C	0.324	45.2
9	Ohio	H M F	0.332	42.9
10	Pennsylvania	H M	0.603	44.4
11	Maine	H C	0.620	44.9
12	West Virginia	H M	0.659	47.4
13	Pennsylvania	H C	0.663	50.0
14	Alabama	H C	0.751	46.4
15	Tennessee	H C	0.956	48.0
16	Missouri	H C	0.956	44.5
17	Pennsylvania	H C	1.01	41.2
18	Michigan	H M	1.23	42.9
19	Ohio	H M	1.25	46.0
20	Tennessee	H C	1.30	47.4
21	West Virginia	H C	1.35	47.4
22	Virginia	H C	1.37	50.0
23	Ohio	H M	1.43	43.6
24	Indiana	H C	1.93	48.0
25	Tennessee	H C	3.26	47.4

[1]H M F—Ohio high magnesium finishing hydrated lime.
[2]H M —High magnesium hydrated lime.
[3]H C —High calcium hydrated lime.
(*a*) Samples received and tested in 1917.
(*b*) Per cent. of water to weight of the paste.

It will be noted that the curve for the lime pastes (Fig. 2) extends downward, from 38 per cent. water to the minimum plastic-

ity value at 48 per cent., and then rises. In other words, the 48 per cent. mixture is the most plastic of the lime pastes. The most plastic lime-gypsum paste however, is that made from a lime paste containing 38 per cent. water or 10 per cent. less than the amount required by the best lime paste. The question arises as to why the plasterer does not use 38 per cent. water instead of 48 per cent. in the preparation of his lime. His practice is probably governed by the fact that a 38 per cent. mixture would be so stiff and unworkable that the increased plasticity gained by using more water would not offset the increased amount of time required in the mixing.

TABLE 2.—EFFECT OF VARIATIONS IN THE WATER CONTENT OF THE PASTES ON THE PLASTICITY AND DENSITY.

Per cent. of water.	"Plasticity figures" of pastes.		Density of pastes.	
	Lime.	Lime-gypsum.	Lime.	Lime-gypsum.
34	...	0.560	1.56	1.52
36	0.705	0.271	1.64	1.53
38	0.780	0.210	1.57	1.53
40	0.709	0.252	1.53	1.49
42	0.567	0.355	1.52	1.49
44	0.527	0.304	1.50	1.49
46	0.454	0.525	1.47	1.51
48	0.447	0.485	1.45	1.48
50	0.486	0.520	1.42	1.48
52	0.527	0.538	1.40	1.48
54	0.675	0.590	1.38	1.46
56	0.695	0.695	1.35	1.45

The densities of the pastes are shown graphically in the lower part of Fig. 2 ("density," as here used, is the weight of a unit volume of the paste in grams per cc.). It will be noted that the densities of the lime pastes decrease uniformly from a value of 1.64 to 1.35 while the densities of the lime-gypsum pastes decrease from a value of 1.52 to 1.45. With an increase of water content the density decreases within the range of the amounts used—the highest plasticity not corresponding to the greatest density.

Clays.—The clays tested were ground to pass a 100-mesh sieve, sufficient water being added to produce the consistency necessary

for jiggering. The clay specimens tested were 3.15″ in diameter and 0.25″ thick. The reduced thickness of the specimen necessitated the use of a flat disk in place of the conical disk. After molding, and previous to testing in the plasticimeter,

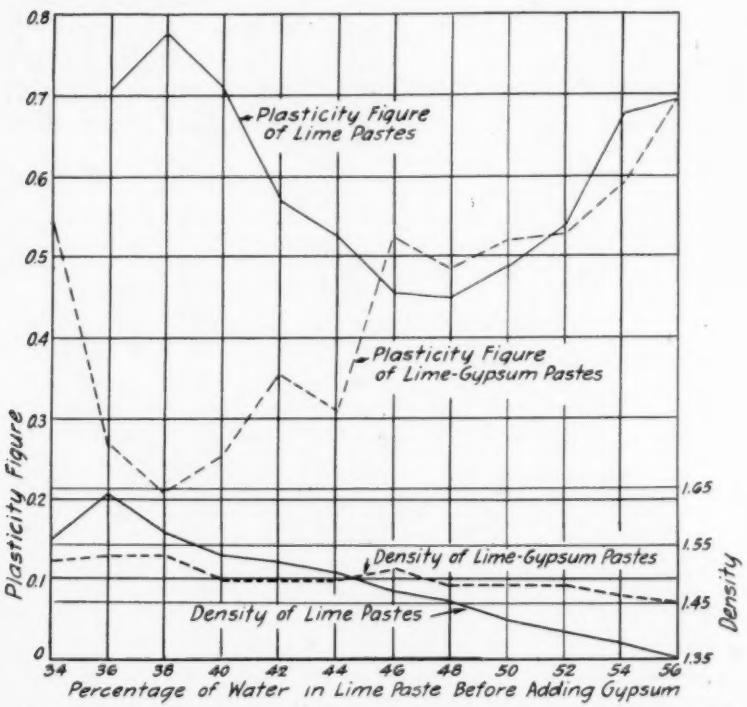


FIG. 2—Effect of consistency of lime paste on plasticity and density of the paste alone and with 25 per cent. gypsum.

the pieces were placed in a moist atmosphere for from 16 to 18 hours. The data secured for the clays is given in Table 3 and is shown graphically in Fig. 3.

It will be noted that the curves for the Tennessee and Kentucky ball clays, which we consider as being very plastic, are similar to those of the plastic limes while the curves of the non-

plastic kaolins are similar to those of the non-plastic limes. The relative behavior of the clays in jiggering appears to be indicated by the character of the curves. The action of the jiggering tool in pressing the clay against the plaster mold is quite similar to that of the plasticimeter disk in pressing the clay against the plaster of Paris block—hence the results should be comparable in either case. Sufficient investigative work has not been done to warrant the claim that the "plasticity figures" represent the relative plasticities of the clays, although they appear to give a comparative indication of the jiggering behaviors of the different clays.

TABLE 3.—"PLASTICITY FIGURES" OF CLAYS AND PORCELAIN BODIES.

Clay	F_5.	F_3.	F_{10}.
Tennessee ball clay	0.397	...	...
Kentucky ball clay	0.561	...	...
Illinois "kaolin"	0.567	...	...
Maryland kaolin	0.600	...	...
Florida kaolin	0.682	...	...
Georgia kaolin	0.790	...	...
New Jersey fire clay	0.879	...	...
North Carolina kaolin	0.992	...	...
English china clay	1.12	...	...
Delaware kaolin	1.37	...	...
Porcelain bodies:			
K1 (not aged)	...	1.05	...
K2 (not aged)	...	0.785	...
K3 (not aged)	...	0.645	...
K4 (not aged)	...	0.622	...
K5 (not aged)	...	0.483	...
No. 26 (not aged)	...	...	0.857
No. 26 (aged 8 months)	...	...	0.623
No. 34 (not aged)	...	...	0.937
No. 34 (stored 8 months)	...	...	0.650

F_5 = 0 to 5 minutes.
F_3 = 0 to 3 minutes.
F_{10} = 0 to 10 minutes.

Porcelain Bodies.—A number of typical porcelain bodies were prepared in the usual manner and tested in the plasticimeter. Some of the bodies were tested at once while others were aged for

8 months previous to the testing. In the preparation of the bodies, sufficient water to afford the desired consistency for jigger-

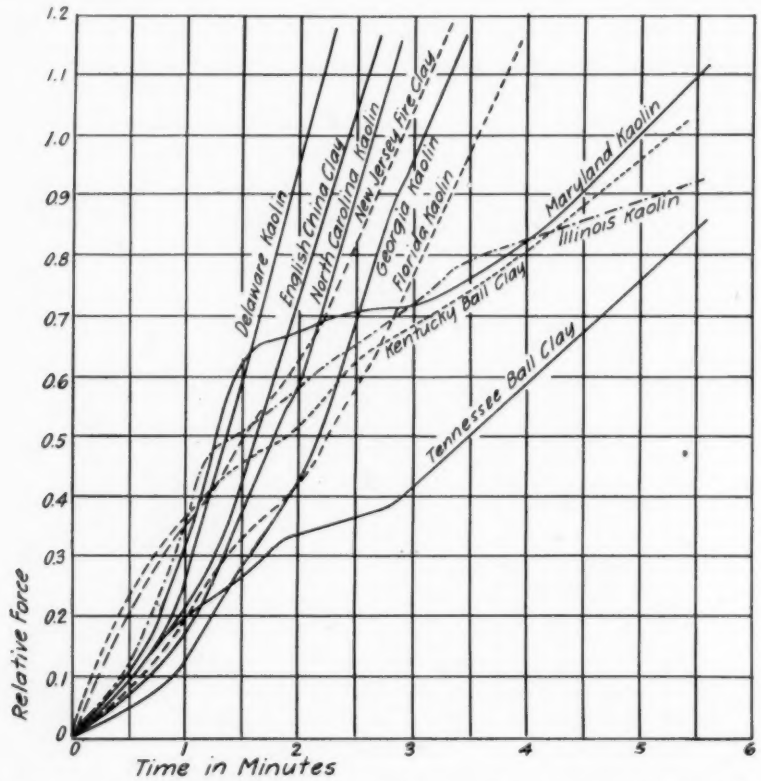


FIG. 3.—Relation between time and relative force for clays in the Bureau of Standards plasticimeter (using flat disk and quarter-inch specimens).

ing was added. The compositions of the bodies were as follows:

Body No. K1:	Per cent.
North Carolina kaolin	15.0
Georgia kaolin	15.0
Delaware kaolin	15.0
Feldspar	20.0
Flint	35.0
	100.0

Body K2—Same as K1 with 4 per cent. Tenn. and Ky. ball clays replacing 4 per cent. of the kaolin.

Body K3—Same as K1 with 8 per cent. Tenn. and Ky. ball clays replacing 8 per cent. of the kaolin.

Body K4—Same as K1 with 12 per cent. Tenn. and Ky. ball clays replacing 12 per cent. of the kaolin.

Body K5—Same as K1 with 16 per cent. Tenn. and Ky. ball clays replacing 16 per cent. of the kaolin.

Body No. 26.	Per cent.
N. Carolina kaolin	12.5
Florida kaolin	12.5
Delaware kaolin	12.5
Georgia kaolin	12.5
Feldspar	20.0
Flint	30.0
	100.0

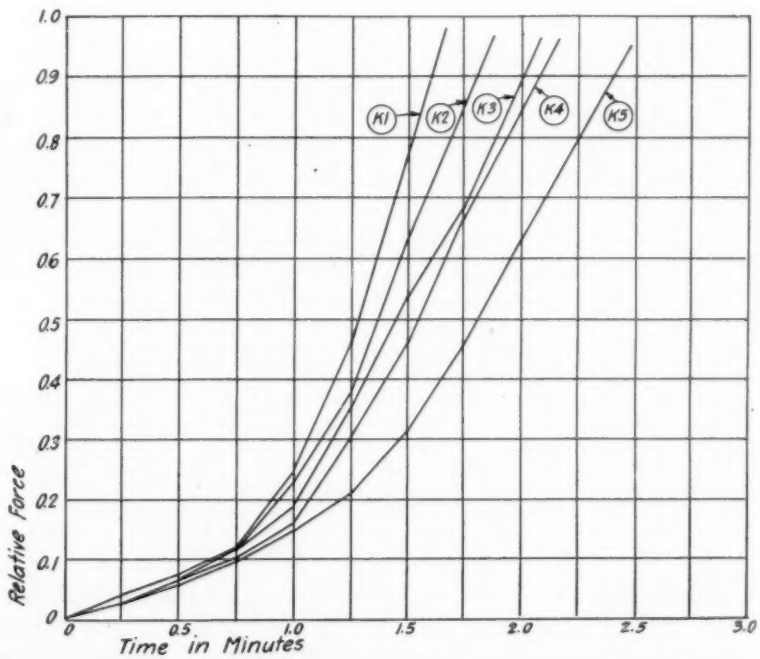


FIG. 4.—Relation between time and relative force for porcelain bodies in the Bureau of Standards plasticimeter (using flat disk and quarter-inch specimens).

Body No. 34.	Per cent.
N. Carolina kaolin	13.75
Florida kaolin	13.75
Delaware kaolin	13.75
Georgia kaolin	13.75
Feldspar	18.5
Whiting	1.5
Flint	25.0
	100.00

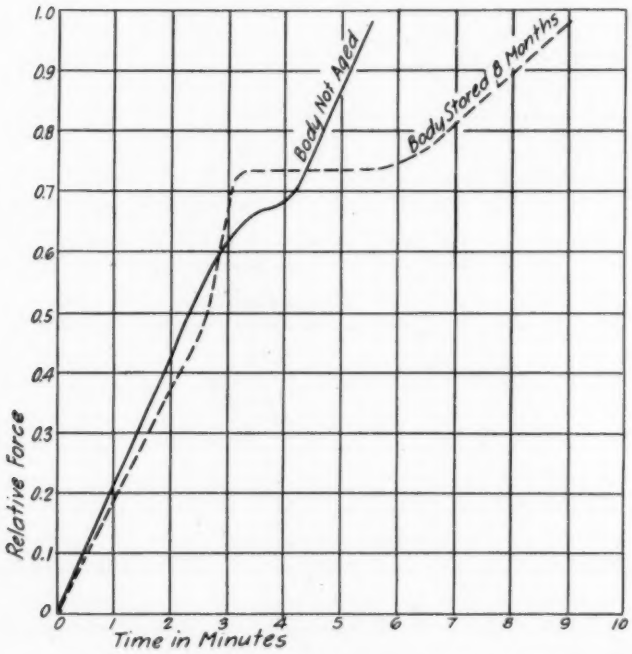


FIG. 5.—Relation between time and relative force for porcelain bodies in the Bureau of Standards plasticimeter (using flat disk and quarter-inch specimens).

The results of the tests upon the porcelain bodies are given in Table 3 and are shown graphically in Figs. 4 and 5. Test pieces 1/4" thick were prepared from bodies K1 to K5, and the "plasticity figure" represents the average relative force for the period

from 0 to 3 minutes. An increase of the ball clay content apparently causes a uniform decrease in the "plasticity figure" with the exception of those for bodies K3 and K4 which are quite close together.

Specimens $^1/_2$" in thickness were prepared from bodies 26 and 34 and the "plasticity figure" calculated for a period of time from 0 to 10 minutes. The results are given in Table 3 and shown graphically in Fig. 5. The ageing of the bodies for 8 months has materially improved their jiggering qualities.

Conclusion.

It should be noted that the determinations on the clays are not numerically comparable with those for the limes since different weights were used on the pendulums in the testing of the two materials.

It may be said that the plasticimeter has given us some practical information in reference to the properties of hydrated limes and of clay bodies. There is room for improvement, however, in the mechanical construction before the instrument may be employed in correlating plasticity with the other properties of the materials.

It remains to be proven whether the property of retaining water and the resistance to deformation under pressure is a measure of the plasticity of a ceramic body when in the plastic condition.

COMMUNICATED DISCUSSION.

W. E. Emley: I want to thank the authors for the above criticism. If the instrument had not been criticized it would in all probability have died a natural death as so many plasticimeters have done before. It has now received a new lease on life, and I hope that further experiments will bring to light the defects in the present design and help to perfect it.

It is unfortunate that the authors have found it necessary to establish certain empirical conditions for the use of the instrument—such as the consistency of the paste, the thickness of the specimen, the duration of the experiment, etc. This makes it difficult, or rather impossible, to compare the results obtained

under different conditions. The fact that the "plasticity figure" has no absolute meaning blocks any attempt to correlate the plasticity, as measured, with any of the other physical properties of the materials. This subject is worthy of thorough and careful investigation. Even though it should be found impossible to design a machine which would measure plasticity in c. g. s. units, the attempt to do so would undoubtedly contribute largely to our present meagre knowledge of this subject.

It is very gratifying to note the definite statement that the instrument is able to differentiate between finishing limes and other limes. This is the purpose for which the instrument was designed. At present the only way of making such a distinction is by actual trial—by having the lime spread on a wall by a plasterer. This method is not satisfactory. It is too laborious and the results depend upon the personal opinion of the laborer—who is frequently not any too intelligent and is apt to be biased. Such results cannot be expressed in black and white for future reference. It is therefore of great benefit to the industry to have an instrument which can measure and express different degrees of plasticity—even though the results are only comparative, and not absolute.

It is very interesting to know that the instrument gives an indication of the behavior of clays in jiggering. The plasticimeter was not designed for this purpose. This would indicate that the instrument may be adaptable to a wider field of usefulness than has as yet been contemplated.

I glean from the present paper that the instrument is, as now designed, capable of practical use in studying the plasticities of limes. This should facilitate and stimulate the redesign of the instrument to overcome the present technical faults. I hope that the authors will continue their experiments along these lines.

THE USE OF FURNACE SLAG AS GROG IN ARCHITECTURAL TERRA COTTA BODIES.

By R. H. Minton, Metuchen, N. J.

One of the most interesting and important ceramic products, architectural terra cotta, has within recent years fallen into some disrepute on account of a few cases of failure in the building, one common type of failure being so-called "dunting." This is unfortunate because terra cotta lends itself to the modern system of steel construction better than any other type of building material.

Very little has been published on this subject. R. L. Clare[1] states that "the first and most important cause of failure is a defective body mixture." He also states that "experiments which we have made indicate that it is much easier to produce fire cracks or cooling cracks by a slight change in the rate of firing and cooling than it is by changing the body composition over a wide range."

In an investigation of the "dunting" of architectural terra cotta, E. C. Hill[2] states that, "fire cracking seems to depend almost entirely on two factors, namely, the composition of the body and the rate and method of cooling. Regarding the composition, it was noted that bodies composed of very sandy clays are much more sensitive to fire cracking than those made of less sandy, more plastic clays." His final conclusion was that "while cooling is of the greatest importance, the composition and density of the body are large factors in the sensitiveness of the body to fire cracking."

Hill is undoubtedly correct in his belief that the body should be controlled by the size and the amount of the grog, rather than by the use of sandy clays, but he seems to have overlooked

[1] *Trans. Am. Ceram. Soc.*, **19**, 593.

[2] Not published.

the question of the *kind* of grog. The kind of grog is undoubtedly of equal importance to the size and the amount.

Some years ago our attention was drawn to the possibility of using furnace slag as grog in terra cotta bodies, the slag being a material of low cost, light in weight, and possessing sufficient resistance to destruction by the elements. Preliminary tests which we conducted with this material were not satisfactory, on account of the inferior quality of the slag selected, but recently our experiments with slag for this purpose have been quite promising.

In our first experiments we used ordinary crushed slag but it was found that granulated slag, on account of its lightness in weight and freedom from iron, etc., offered greater possibilities. It was soon determined that we were limited to the use of acid slags, the basic slags being unsatisfactory as grog on account of their excessive weight and their content of free lime which would probably cause disintegration of the burned terra cotta bodies. The following two bodies are typical of those prepared in our investigation:

Body No. 174.

Stoneware clay	15 parts (by vol.)
Retort clay	15 parts (by vol.)
Slag	20 parts (by vol.)
$BaCO_3$	$^1/_2$ per cent.

Body No. 175.

Stoneware clay	15 parts (by vol.)
Retort clay	15 parts (by vol.)
Grog (sagger)	20 parts (by vol.)
$BaCO_3$	$^1/_2$ per cent.

A granulated slag from Coatesville, Pa. having the following composition was used in Body No. 174:

	Per cent.
SiO_2	44.1
Al_2O_3	24.1
Fe_2O_3	1.3
CaO	20.6
MgO	6.7
MnO	3.0
	99.8

The bodies were prepared in accordance with the usual terra cotta practice. Shrinkage bars, $14'' \times 3'' \times 3''$, and also sanitary tray legs for glazing tests were molded from the plastic clay. The tray legs were slipped and glazed with a terra cotta slip and glaze maturing between cones 4 and 5.

The test pieces were burned in a coal-fired test kiln with cone 5 turning at the end of a firing period of 45 hours. At cone 5, Body No. 174 develops a dark buff color and Body No. 175 a light buff color. Both bodies develop a good dense structure when fired to this cone.

The absorptions of Nos. 174 and 175 (after immersing for 48 hours in water) were 9.4 per cent. and 12.4 per cent., respectively. The total drying and burning shrinkage of the two bodies, Nos. 174 and 175, was 14.3 and 14.7 per cent., respectively. At the expiration of one year, no signs of shivering, crazing or other defects were noted in the white matt glaze applied to the test pieces.

Freezing Tests.

In order to best determine the relative abilities of the two bodies to withstand weathering action, some of the shrinkage bars from bodies Nos. 174 and 175 were subjected to a so-called artificial freezing test as follows:

After boiling for three hours in a sodium sulphate solution, the test pieces were removed and dried at room temperature for 48 hours—the immersion and boiling treatments being repeated a number of times. No apparent effect of this treatment was observed on either body up to the fifteenth test, after which Body No. 175 began to flake slightly. Body No. 174 was not affected after being subjected 34 times to the treatment. The condition of the two bodies at various stages of the treatment was noted as follows:

After 20th treatment—B 175, flaking on all surfaces.....B 174 not affected.
After 24th treatment—B 175, entire surfaces on four sides scaling off.....B 174 not affected.
After 29th treatment—B 175, scaling quite rapidly.....B 174 not affected.
After 34th treatment—B 175, scaling off in large, thin flakes.....B 174 not affected.

The condition, after the 34th treatment, of two of the shrinkage bars was as shown in Fig. 1. It will be noted that Body No. 174 was practically unaffected by the sodium sulphate test while Body No. 175 is partly disintegrated and has the appearance of a rock-faced building brick.

FIG. 1.—Bodies Nos. 174 and 175 after 34 sodium sulphate treatments.

In order to determine the resistance of the two bodies to actual freezing, pieces of each were immersed in water until saturated, thoroughly frozen at 5° F., and then thawed. Both bodies were apparently unaffected after having been frozen and thawed twenty-five times. The compressive strength of the two bodies, before and after the natural freezing, was as follows:

	Compressive strength, pounds per sq. in. (Before freezing.)	Compressive strength, pounds per sq. in. (After freezing.)
Body No. 174	6710	6410
Body No. 175	5900	3660

It will be noted that the freezing treatments have affected but slightly the compressive strength of Body No. 174, whereas the compressive strength of Body No. 175 was reduced about 40 per cent.

Experimental Bodies.

In order to compare the slag type of body with others, a number of mixtures, in which were used sagger grog, slag, slag and sagger

grog mixtures, and vitrified porcelain grog as the non-plastic ingredients, were prepared as follows:

Body No. 200—commercial terra cotta body
" No. 201—commercial terra cotta "
" No. 202—commercial terra cotta "
" No. 203—Stoneware clay 15 parts (by vol.)
Retort clay 15 parts (by vol.)
Grog (sagger) 20 parts (by vol.)
$BaCO_3$ $^1/_2$ per cent. by weight
" No. 204—Stoneware clay 15 parts (by vol.)
Retort clay 15 parts (by vol.)
Grog (sagger) 10 parts (by vol.)
Granulated slag 10 parts (by vol.)
$BaCO_3$ $^1/_2$ per cent. by weight
" No. 205—Same as Body No. 204 but containing 20 parts of slag
" No. 206—Same as No. 204, but containing 20 parts of electrical porcelain grog
" No. 207—Stoneware clay 15 parts (by vol.)
Retort clay 15 parts (by vol.)
Grog (sagger) 16 parts (by vol.)
Granulated slag 4 parts by (vol.)
$BaCO_3$ $^1/_2$ per cent. by weight

The granulated slag used in Bodies Nos. 204 and 205 had the following composition:

	Per cent.
SiO_2	32.4
Al_2O_3	12.2
CaO	39.3
MgO	11.6
MnO	0.8
S	1.8
	98.1

The slag used in Body No. 207 was from the Illinois Steel Co., the composition being as follows:

	Per cent.
SiO_2	34.2
Al_2O_3	12.4
CaO	47.5
MgO	3.6
Fe_2O_3MnO	0.9
S	1.7
	100.3

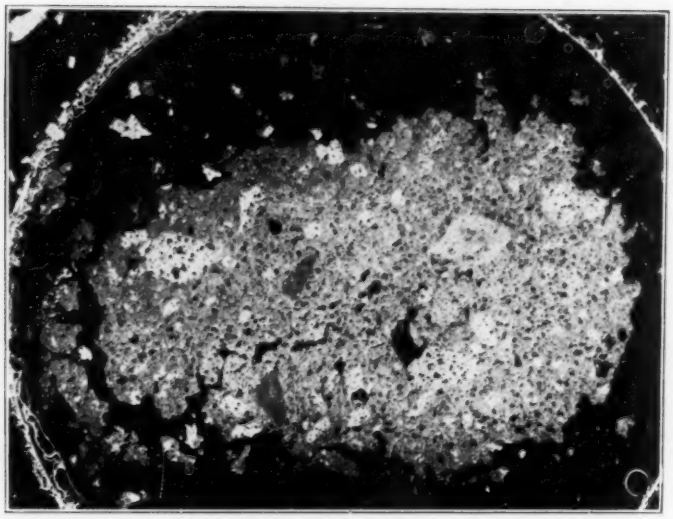


FIG. 2.—No. 202, Commercial terra cotta body.

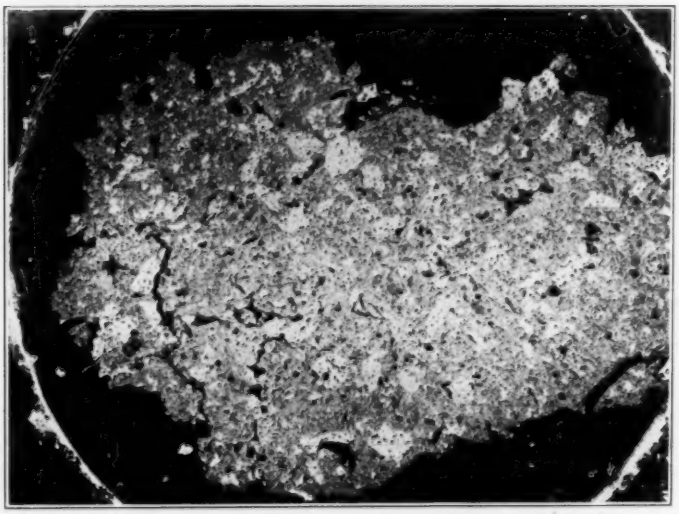


FIG. 3.—No. 203, Experimental body, similar in composition to commercial body.

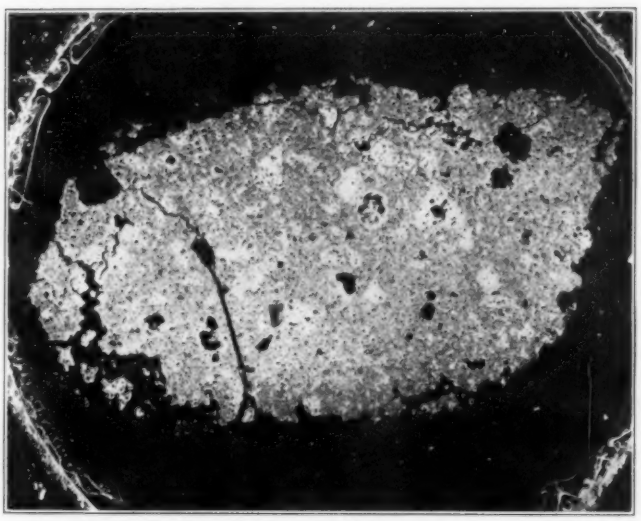


FIG. 4.—Body No. 205, containing 20 per cent. slag as grog.

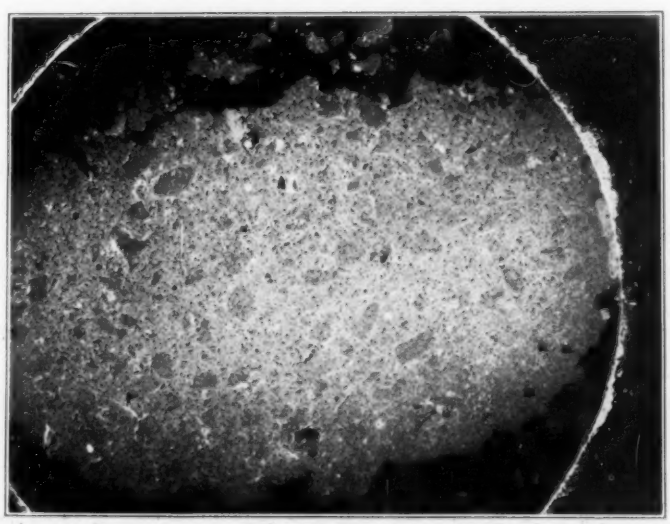


FIG. 5.—Body No. 206, containing 20 per cent. crushed porcelain as grog.

In Figs. 2, 3, 4 and 5 is shown the structure afforded by the different types of grog in Bodies Nos. 202, 203, 205 and 206, respectively. The photographs were taken of a section of each of the bodies after grinding to a smooth surface on a carborundum grinder.

The shrinkage, absorption, and crushing strength of each body was found to be as follows:

Body No.	Per cent. burning. Shrinkage. (Basis dry length.)	Per cent. absorption. Burned to Cone 4–5.	Crushing strength. Burned to Cone 4–5.
200	3.5	10.9	.....
201	2.9	11.1	4600 lbs. per sq. in.
202	4.9	11.9	.....
203	3.9	11.0	3250 lbs. per sq. in.
204	4.9	7.2	4325 lbs. per sq. in.
205	5.0	10.1	3490 lbs. per sq. in.
206	5.9	7.6	5300 lbs. per sq. in.
207	3.5	13.4	3325 lbs. per sq. in.

The results of the sodium sulphate tests made on these bodies may be summarized as follows:

After 50 treatments.

No. 200—Scaling considerably at one end.
201—Scaling somewhat.
202—Scaling slightly at one end.
203—Flaking considerably on edges.
204—Very slightly affected on bottom surface and edges.
205—Only slightly affected.
206—Flaking on the edges.
207—Flaking considerably at one end.

The treatments will be continued until complete disintegration has taken place. After fifty treatments bodies Nos. 205 and 206 were the least affected. It was to be expected that a body containing electrical porcelain, stoneware, or a similar vitrified grog would be more resistant than one containing fire-clay grog. However, the vitrified grog is not obtainable in large quantities is heavier, and detracts from the working properties of a body.

Conclusion.

If further experiments confirm the practicability of the use of slag as grog in architectural terra cotta, an abundant material

at less than one-half the cost of sagger grog, and also considerably lighter in weight, is available. In the selection of a slag, careful attention must be given to the source and quality—we have found the granulated slag to be the best for this purpose. In the above experiments we have experienced little difficulty from the presence of iron in the slag but this is a matter which would require some attention. Any iron present may possibly be removed by passing the slag over a magnetic pulley.

This opportunity is taken to express our appreciation for the assistance of Mr. E. Ogden in making the freezing and crushing strength tests.

COMMUNICATED DISCUSSIONS.

P. H. SWALM: To the many theories for the causes of "dunting" Mr. Minton's investigation adds one to be well considered. Is the nature of the grog a factor in "dunting?" Unfortunately, for lack of better tests, his conclusions are based upon the disintegration or flaking of the body and his crushing strength data. Is it not a fact that the stronger bodies which resist disintegration are also subject to the fault of sudden rupture or "dunting?" This has been my experience with fire clay sanitary ware bodies.

It is possible to understand why a body gives way under rapid cooling in the kiln, and it is to be expected that a dense body, improperly bonded, might "dunt" under strain at some later time, but it seems to me that the "dunting" of a well bonded strong body at a delayed period may only be caused, either by a defect in molding or by the unusual physical properties of one of the ingredients of the body. By the use of tight burning, sandy, heavy fire clays, I have produced fire clay sanitary bodies of apparently good quality, but found that these bodies "dunt" very readily, regardless of the size, quantity, and vitrification behavior of the grog used. This was probably due to the introduction of an excessive amount of silica as like results were secured by the substitution of coarse sand for the fire clay grog.

The properties of the grog must therefore be considered. Mr. Minton's investigation shows that by the use of slag a stronger and more resistant body is produced but the effect on "dunting"

is not conclusively proven. I have found that the use of electrical porcelain grog improved the body, but I could not entirely eliminate the sagger grog from the mixture on account of the reduction in strength in the raw clay state. The porcelain grog, being vitrified, is not conducive to a firm bond between the grog and the clay. The latter point is one to be well considered in the use of the slag as grog in fire clay bodies.

H. W. MOORE: The terra cotta companies have, for at least two years back, been trying to improve the quality of their bodies and the use of slag as suggested by Mr. Minton is, so far as I know, something entirely new. I have tried a sample of slag myself, but found it unsuitable for this purpose. If, by the use of the slag from the Midvale Steel Company, it is possible to produce a body which will successfully resist the sodium sulphate tests as applied by Mr. Minton, obviously, a great stride in the improvement of terra cotta bodies has been accomplished, as, in my opinion, such a body would withstand any atmospheric exposure to which it would be subjected.

E. C. HILL: Mr. Minton, in advocating the use of furnace slag as grog in architectural terra cotta, has introduced a material that is new to the industry, but which may prove of considerable importance in the future. If slag is available at one-half the cost of broken saggers, as Mr. Minton states, and can be obtained free from impurities and constant in composition, its use is certainly worth considering.

Mr. Minton refers to failures of terra cotta due to "dunting" and suggests as a remedy the use of slag as a source of grog instead of saggers. The results show that the slag body has a much lower absorption and greater crushing strength than the sagger body and successfully withstands the artificial freezing test with the sodium sulphate solution while the sagger body fails in this test. His conclusions, based on this test, are that the slag and vitrified porcelain bodies are more able to withstand the action of the weather than the sagger body, although he attributes the failure of terra cotta in practice to be chiefly due to "dunting."

The failure of terra cotta from "dunting" is due to its inability to withstand changes of temperature, involving the expansion and contraction of the piece, and not to its inability to withstand disintegration or flaking off, due to the destructive action of freezing and thawing. When terra cotta fails from "dunting" the piece is broken or cracked in sharp lines, the fracture being smooth and cleancut; when it fails from disintegration, due to freezing and thawing, the failure is evidenced by crumbling of the edges and the flaking off of the sides.

The character of the failure produced by the artificial freezing test, crumbling and flaking of the piece, would be expected from the continued action of natural freezing and thawing but I have never observed the sharp hair-line cracks, so characteristic of "dunting," in any of the test pieces subjected to the sulphate treatment. I do not think that "dunting" cracks could be produced by this treatment unless the pieces were quite large, in which case a failure would be more likely to result from the continued rapid heating and cooling than from the disruptive action of the crystallized salt from the sulphate treatment.

Bureau of Standards Report.

In this connection, I would quote the following from a report of the Bureau of Standards, dated May 10, 1918, to the National Terra Cotta Society, concerning an investigation of terra cotta bodies: "The value of this test (artificial freezing) as a criterion of the weather-resisting properties of a body is open to some question. The appearance of the disintegrated specimens closely resembles that shown by terra cotta which has failed in service, due to alternate freezing and thawing, although similarly prepared specimens have been frozen and thawed 37 times during the past winter and do not show at all the same behavior. Only 8 cubes (4″ solid) show any effect of the natural freezing and it consists of a clean, sharp break of the cube into two fragments. None of the open boxes (8″ × 8″ × 6″ deep with 1″ walls) show effects of any kind caused by the same number of freezings. According to Staley,[1] one sodium sulphate treatment of clay drain tile is equivalent to two natural freezings, if the tile in the

[1] *Trans. Am. Ceram. Soc.*, **18**, 642–923 (1916).

latter case are partly immersed in the water while freezing. In the present investigation no such ratio has been obtained for some of the specimens disintegrated at the end of two sodium sulphate treatments but were intact at the end of 37 natural freezings, a ratio of 1 to 18. The terra cotta specimens were, however, not immersed during the freezing but were saturated with cold water."

Theory of "Dunting."

(E. C. HILL.)

Terra cotta bodies giving the best results in the artificial freezing tests are those that are hard and dense and having comparatively low absorption and high compressive strength. A body of this kind would, in all probability, be more sensitive to "dunting" than one less dense and would have a correspondingly higher absorption and lower strength. In a paper read by the writer before the New Jersey Clay Workers' Association, June, 1917, on fire cracking or "dunting" in terra cotta, from which Mr. Minton has quoted, the causes of "dunting" were discussed as follows: "The failure due to "dunting" is evidenced by sharp hair-line cracks in the piece either occurring in the kiln or appearing later when set in the building. The cracks are more liable to occur on large pieces of heavy cross-section than on the smaller, lighter pieces. When the cracks appear in the building they are more likely to be found on pieces which are exposed on all sides than on those set in the wall. The cause of the failure by "dunting" is attributed to strains set up in the body during cooling. These strains produce the hairline cracks at the time, or cause cracking later when the pieces are subjected to changes in temperature. Experiments made show that fire-cracks may be produced in all bodies by rapid cooling and that, if cooled slowly enough, all bodies are free from the fire-cracks.

Regarding the composition of the body, it appears that the hard, dense bodies are more sensitive to fire-cracks as are also those containing very sandy clays or highly siliceous fire brick grog.

With reference to the effect of silica in the clay and grog, data submitted by Purdy[1] on the expansion and contraction of

[1] *Trans. Am. Ceram. Soc.*, **15**, 499–522 (1913).

porcelain body mixtures, shows that the expansion and contraction of fired test pieces, heated to and cooled from 900° C, is the greater the higher the flint content, and that the rate of expansion and contraction between 650° and 500° C is much greater than during other stages and that the higher the flint content, the greater is the rate of expansion and contraction during this period. The limits of composition as employed in Purdy's investigation with bodies containing clay and flint was 20 per cent. clay, 80 per cent. flint and 90 per cent. clay and 10 per cent. flint. Purdy noted that the total expansion at 900° C of the high flint body is about $2^1/_2$ times that of the high clay body and that the rate of expansion and contraction between 600° and 500° C of the high flint body is about 7 times as great as the high clay body, and for temperatures below 500° C about 3 times as great. Data obtained in the same way by Boeck,[1] on fired test pieces of fire clay and ball clay, shows that the expansion and contraction curve for fire clay has the same general trend as the porcelain bodies containing flint in Purdy's investigation, but the ball clay has a comparatively uniform rate of expansion and contraction between 900° and 200° C.

If, in cooling a high clay body, the contraction takes place at a uniform rate over a considerable temperature range, the body becomes more sensitive to strains as its rigidity increases, so that strains are more likely to occur at a dull red heat and after, than at the higher temperatures. When a body contains sandy clays, however, it not only has to contract more in cooling, but has to withstand the greatest rate of contraction during the period within which it is most sensitive to cooling strains.

Since it appears that the very dense, hard bodies are more sensitive to "dunting" than the more open porous ones and since it is common practice in manufacturing many clay wares to control the density, either wholly or in part by the introduction of sandy clays, it is suggested that the density of terra cotta bodies be controlled as far as possible by the size and amount of the grog, rather than by the use of sandy clays. It is further suggested that the cooling be conducted slowly below 650° C.

[1] *Trans. Am. Ceram. Soc.*, **14**, 470–479 (1912).

While the method and rate of cooling is of the greatest importance in preventing "dunting," and terra cotta bodies of almost any density or composition will be free from dunting if cooled slowly enough, it is possible that, with the ordinary rate of cooling, in attempting to compound a body with low absorption, high crushing strength, and which will stand up best in the artificial freezing test, we might arrive at one that would be quite sensitive to "dunting"—particularly when manufactured into large pieces which are exposed on all sides to the action of the weather.

In order to manufacture terra cotta with straight and true lines it is essential that the body have as low a drying and burning shrinkage as possible, and that there be no tendency to warp in firing. These requirements are met with a body having a somewhat open, porous structure, rather than one which is hard and dense.

The body for terra cotta should have a reasonably low absorption, high crushing strength and stand up reasonably well in the artificial freezing test, but it is not advisable to go beyond this on account of the increased shrinkage, tendency to warp in firing, and sensitiveness to "dunting." In order to secure these desired properties I do not believe that it is necessary to develop a body which is able to withstand 30 of the sodium sulphate tests.

R. H. MINTON: Mr. Swalm admits my contention that the "character" of the grog and clays used is a more important factor in producing "dunting" than any other. This particularly applies to sandy grog or clays. His objection to the use of vitrified grog—owing to the difficulty of bonding with the plastic clays, is not sustained, as many terra cotta manufacturers have used vitrified grog entirely for certain purposes, and other manufacturers use it in large quantities. I have not experienced this difficulty in using vitrified grog, and even so, it would not arise in the use of the slag which does not have the smooth, clean surfaces of the porcelain and stoneware grogs.

It is perhaps unfortunate that my conclusions were based upon the disintegration and crushing strength data, but it is not clear

what other tests could have been used—as those employed are most conclusive in the determination of the weather-resisting qualities of any clay product.

Mr. Hill states that "the failure of terra cotta from 'dunting' is due to the inability to withstand changes of temperature—involving the expansion and contraction of the piece—and not to its inability to withstand disintegration or flaking due to the destructive action of freezing and thawing." Mr. Hill has not proven that "dunting," which develops in the wall, is not produced by the destructive action of freezing and thawing, regardless of what may be the primary cause. However, it was not intended to attempt to produce actual "dunting" by the freezing tests. These tests and the crushing tests were applied because they appeared to be best adapted to a measurement of the resistance of clay bodies to weathering action.

Mr. Hill further states that "terra cotta bodies giving the best results in the artificial freezing tests are those that are hard and dense and having a comparatively low absorption and high compressive strength; a body of this kind would, in all probability, be more sensitive to 'dunting' than one less dense and having a correspondingly higher absorption and lower strength." Mr. Hill seems to overlook the real point at issue. The use of slag, or vitrified grog, does not necessarily produce a denser body so far as the clay matrix is concerned. The absorption is lowered merely because the slag grog has a lower absorption than the porous sagger grog. One of the terra cotta manufacturers has for many years used vitrified chemical stoneware or electrical porcelain grog in the manufacture of terra cotta which he supplies to a Canadian customer. Another manufacturer considered the use of common stoneware for the same purpose.

Mr. Hill also states: "it appears that the hard, dense bodies are more sensitive to fire-cracks than the others as are also those containing very sandy clays or highly silicious fire brick grog." And again: "since the very dense, hard bodies seem more sensitive to 'dunting' than the more open porous ones, and since it is a common practice in the manufacture of many clay wares to control the density, either wholly or in part by the introduction of sandy clays, it is suggested that the density of terra cotta

bodies be controlled as far as possible by a variation in the size of the grog, rather than by the use of sandy clays."

In his original paper on "dunting" he states: "it has been noted that the bodies containing sandy clays are more sensitive to fire cracking than those made up of the more plastic clays." Also: "fire cracking seems to depend almost entirely on two factors, namely, the composition of the body and the rate and method of cooling. Regarding the composition, it was noted that bodies composed of very sandy clays are much more sensitive to fire cracking than those made of less sandy, more plastic clays." These statements seem to be contradictory.

My contention is that the "dunting" and failure of the terra cotta is dependent more upon the "character" or "quality" of the grog and clays than upon the other factors and that a grog of low absorption is superior to the porous sagger grog. So far as price, weight, availability, working properties, etc., are concerned, it would appear that the slag is to be preferred to the grogs prepared from porcelain, stoneware, etc.

THE EFFECT OF ELECTROLYTES ON SOME PROPERTIES OF CLAYS.[1]

By H. G. SCHURECHT, Columbus, Ohio.

Introduction.

One of the sources of loss in the manufacture of clay products, especially of the larger kind, chemical stoneware, glass pots, etc., is due to the lack of strength of the clays in the dry state and at the burning temperatures. Sufficient strength to resist the shocks and strains of handling in the green state is necessary. At certain periods in the burning the ware loses strength and is liable to warp, crack, or break, causing large kiln losses. The natural remedy would be to improve the strength of the clay body by artificial means or to introduce stronger clays.

The effect of the addition of organic compounds to clays has been studied by Acheson,[2] Minton[3], Seger and Cramer,[4] and Koerner,[5] and it has been shown that the strength of the clays can be increased by the addition of gallo-tannic acid, catechu, straw emulsion, dextrine, or starch.

The effect of the addition of alkalies and acids on the volume shrinkages of clays in the plastic state has been reported by Bleininger and Fulton,[6] and Kerr and Fulton,[7] but our information as to the effect of these electrolytes on the strength of clays is meagre. Bleininger[8] has reported that cast bodies containing alkalies may be stronger than bodies not containing alkalies. The writer[9] has noted that the rate of disintegration in water is much slower for the deflocculated than for the flocculated clays.

[1] By permission of the Director, Bureau of Mines.
[2] *Trans. Am. Ceram. Soc.*, **6**, 31–64 (1904).
[3] *Ibid.*, **6**, 231–259 (1904).
[4] *Wein und Leipzig, A. Hartleben's Verlag*, **1909**, p. 82.
[5] *Op. cit.*, pp. 82–84.
[6] *Trans. Am. Ceram. Soc.*, **14**, 827–839 (1912).
[7] *Ibid.*, **15**, 184–192(1913).
[8] Bureau of Standards, *Tech. Paper* **51**, 40 (1915).
[9] *Trans. Am. Ceram. Soc.*, **19**, 144 (1917).

As the rate of its disintegration is sometimes used as a rough indication of the bonding power of a clay, further investigations with a view to studying the effect of the addition of alkalies and acids on the strengths of clays were carried out. A surprising increase in strength was gained by the addition of the alkalies, in some cases the strength of the treated clays being four times as great as that of the untreated. The present investigation was limited to a study of the effect of the addition of alkalies and acids on the properties of clays with special reference to their strength.

Method of Testing.—For the investigation the following mixtures were selected:

(1) 50 parts Kentucky ball clay and 50 parts flint.
(2) 50 parts Tenn. ball clay and 50 parts flint.
(3) 50 parts Georgia kaolin and 50 parts flint.
(4) 50 parts Kentucky ball clay and 50 parts grog.
(5) 100 parts Kentucky ball clay.

The cross-breaking strength, water of plasticity, drying shrinkage, density and porosity were determined for each clay in the raw condition. The tests were made on the untreated clays and on the clays to which 0.05, 0.10, 0.15, 0.20, 0.40, 0.60, 0.80, 1.00, 1.50, 2.00, 3.00 and 5.00 per cent. (by weight) of electrolyte had been added. The electrolytes added were sodium hydroxide, sodium silicate, sodium carbonate, calcium hydroxide, tannic acid, and sulphuric acid.

Tests of the Clays in the Raw Condition.—For the cross-breaking tests, bars 1″ x 1″ x 7″ were molded and dried at room temperature for 3 days, then in a drying oven at 65° C for 24 hours, and finally in the oven at 110° C for 24 hours. A span of 5 inches was used in breaking the pieces transversely. The modulus of rupture of each piece was calculated—the average of ten determinations being taken as the final result.

In making the water of plasticity and the drying shrinkage determinations, the electrolyte was added to a quantity of water slightly less than that required for developing the maximum plasticity. In case the limits for the normal consistency were large, the water was added by volume so as to keep the per cent.

added as nearly as possible the same, thereby making the shrinkage data more comparable.

The drying shrinkage was determined by the use of the kerosene immersion method and the per cent. volume shrinkages were calculated on the basis of the dry volumes.

The per cent. shrinkage in terms of the true clay volume was determined by the method described by Bleininger[1] as follows:

$$s = \frac{d(v_1 - v_2)}{w} 100.$$

s = per cent. volume shrinkage in terms of the true clay volume.
d = specific gravity of the powdered clay.
v_1 = volume of the clay briquette in the wet condition.
v_2 = volume of the clay briquette in the dried condition.
w = weight of the dried briquette.

Tests of the Clays after Burning.—The test pieces were burned to cone 01 in a gas-fired test kiln, the temperature being increased at the rate of 30° C per hour from 1000° C to within 20° C. of the finishing temperature—at which point the rate was reduced to 10° C per hour. The apparent porosity, volume shrinkage, and density of each briquette was determined.

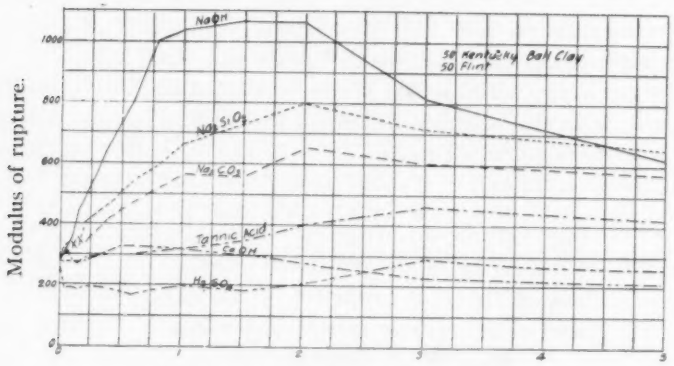
The cross-breaking tests were made on $4'' \times 0.8'' \times 0.8''$ bars, using a span of $3.2''$.

Effect of the Electrolytes on the Properties of the Clays and Mixtures.

Dry Strength.—In Figs. 1 and 2 is shown the effect of the addition of the electrolytes on the dry strength of the clays and mixtures studied. The addition of the alkalies and tannic acid to the clays increased the strength while the addition of sulphuric acid decreased the strength. Calcium hydroxide, when added in small quantities (0.2 to 2.0 per cent.), improves the strength somewhat but weakens the bodies when added in larger amounts. The maximum effect is produced by the addition of from 0.8 to 2.5 per cent. of the alkalies, or about 3.0 per cent. of tannic acid.

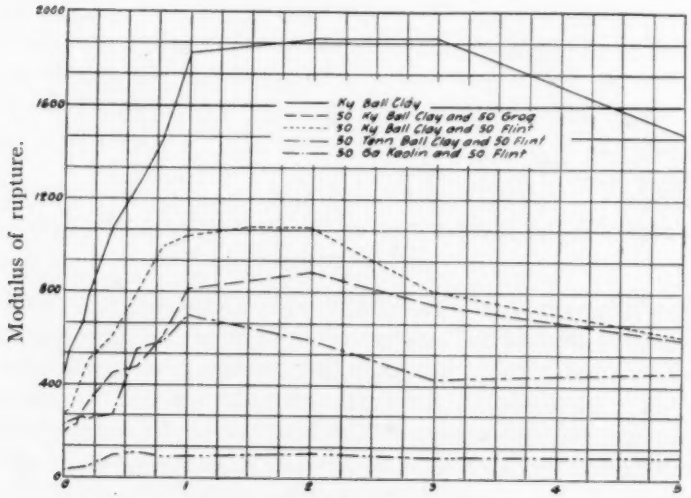
[1] Bureau of Standards, *Tech. Paper* 1, 21 (1910).

When caustic soda was added the bodies were molded with some difficulty and would not retain their shape unless a low per cent.



Per cent. electrolyte in terms of clay.

FIG. 1.—Dry Strength.



Per cent. NaOH in terms of clay.

FIG. 2.—Dry Strength.

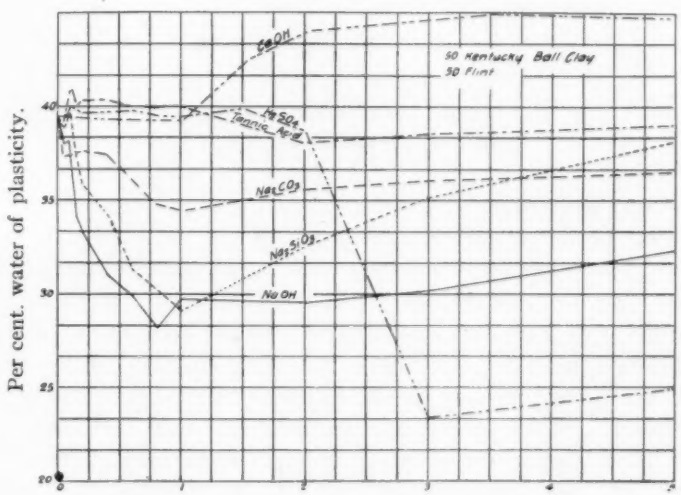
of water was employed, although in this condition the clays laminated badly. This effect was most pronounced in the mixtures

developing the greatest strength (see Fig. 1). The addition of sodium silicate, sodium carbonate, or more than 2 per cent. of sulphuric acid, affected the molding properties to a smaller extent and the addition of calcium hydroxide or tannic acid had little effect on the molding properties. The addition of less than 2.0 per cent. sulphuric acid was found to improve the working properties of a body.

The natural brown color of the ball clays changed to black upon the addition of a large percentage of alkalies or acids. The use of calcium hydroxide did not materially affect the natural coloring of the clays although it did cause the formation of flaky scales on the surfaces of the test pieces.

Under the microscope the clays treated with the alkalies appeared to be very fine grained, indicating that the clay substance present was in a deflocculated condition.

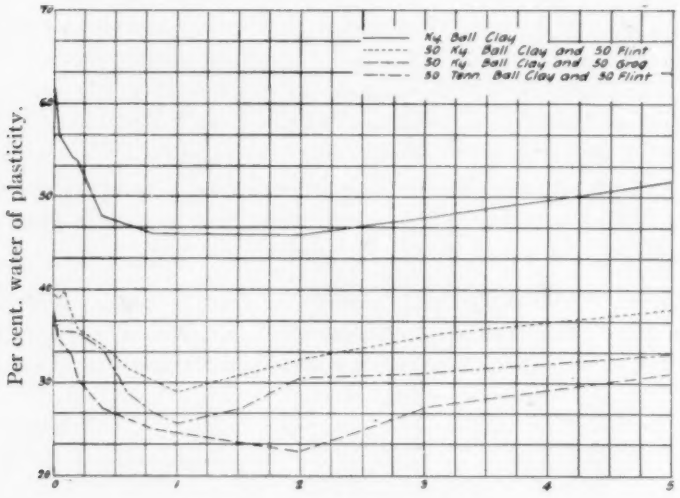
Water of Plasticity.—The curves in Figs. 3 and 4 show the effect of the electrolytes on the per cent. water of plasticity. In the case of the two ball clays the addition of the alkalies decreases



Per cent. electrolyte in terms of clay.

FIG. 3.—Water of Plasticity.

the per cent. water necessary to the development of the plasticity. The addition of a small amount of acid necessitates the use of a greater amount of water in working the clays but, if large quantities are added, a smaller amount of water must be used. The addition of calcium hydroxide increases the per cent. water of plasticity.

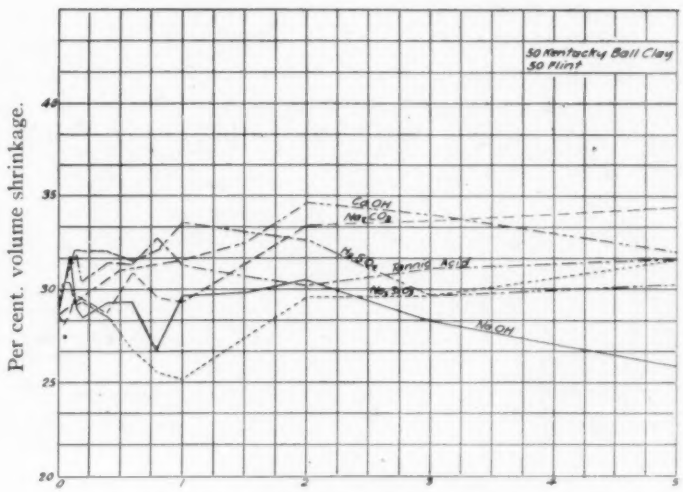


Per cent. NaOH in terms of clay.

FIG. 4.—Water of Plasticity.

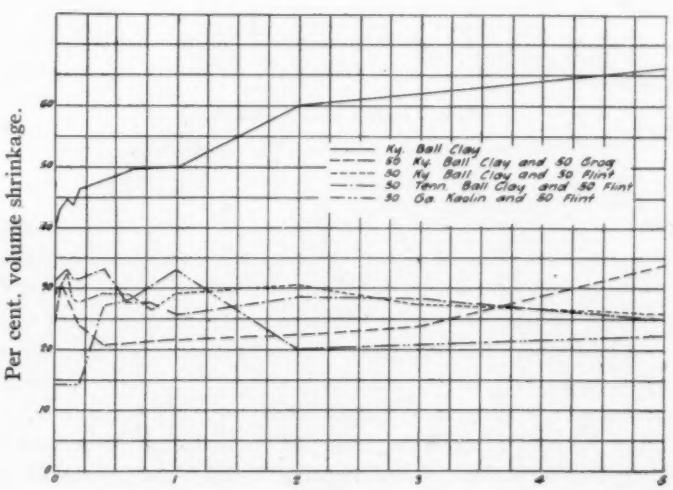
Volume Shrinkage.—In Figs. 5, 6 and 7 is shown the effect of the electrolytes on the volume shrinkages of the clays and mixtures. The addition of one per cent. of alkalies appears to decrease the volume shrinkage of the mixtures of 50 parts clay and 50 parts flint with the exception of the one containing Georgia kaolin, the shrinkage of this mixture being increased. The addition of sodium hydroxide to the Kentucky ball clay increases the shrinkage. Small percentages of sulphuric acid increase and large percentages decrease the volume shrinkages. The addition of calcium hydroxide and tannic acid increases the volume shrinkage.

The ratio of pore to shrinkage water becomes lower as the per



Per cent. electrolyte in terms of clay.

FIG. 5.—Volume Shrinkage.



Per cent. NaOH in terms of clay.

FIG. 6.—Volume Shrinkage.

cent. of shrinkage water in terms of the total water increases until 2.0 per cent. of caustic soda has been added when the reverse is true. As clays having a low pore to shrinkage water ratio and a high per cent. of shrinkage water in terms of the total water often developed good strength, these results are in accordance with the strength data. (See Fig. 1.)

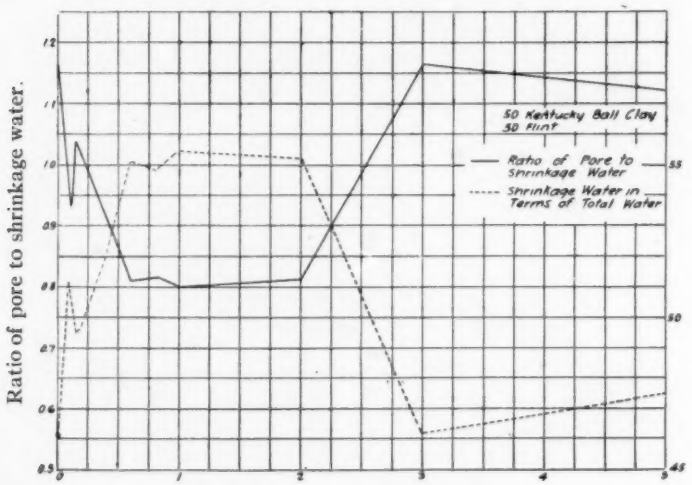
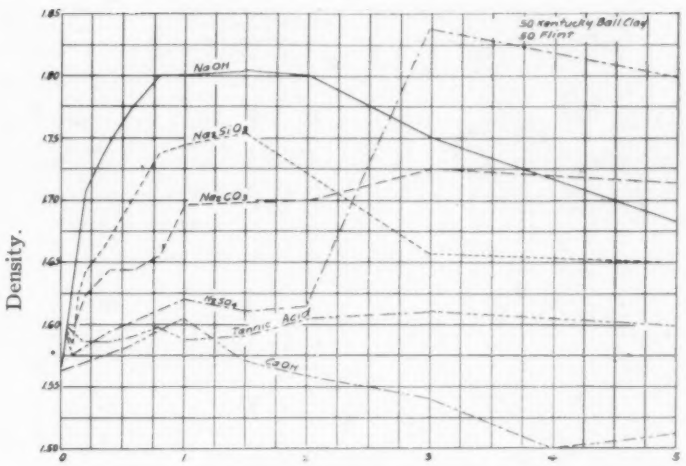

Ratio of pore to shrinkage water.
50 Kentucky Ball Clay
50 Flint
Ratio of Pore to Shrinkage Water
Shrinkage Water in Terms of Total Water
Per cent. NaOH in terms of clay.

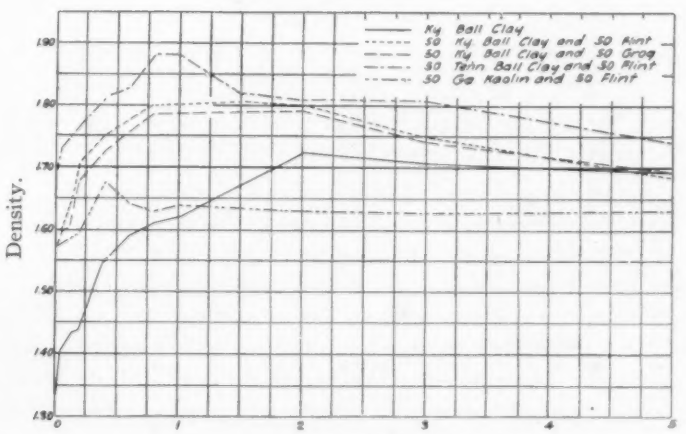
FIG. 7.—Shrinkage and Pore Water Relation.

Density.—In Figs. 8 and 9 are shown the effect of the electrolytes on the densities of the clays and mixtures. The alkalies and tannic acid increase the densities of the bodies, the maximum effect being produced by the addition of from 0.40 to 3.0 per cent. When more than 2.0 per cent. sulphuric acid is used the density is increased to a remarkable extent. However, there is only a small increase in strength (see Fig. 1), which may be accounted for by the oxidizing effect of the sulphuric acid and the consequent destruction of the organic bond. Additions of calcium hydroxide up to 1.0 per cent. increases the density. The addition of larger amounts decreases the density.



Per cent. electrolyte in terms of clay.

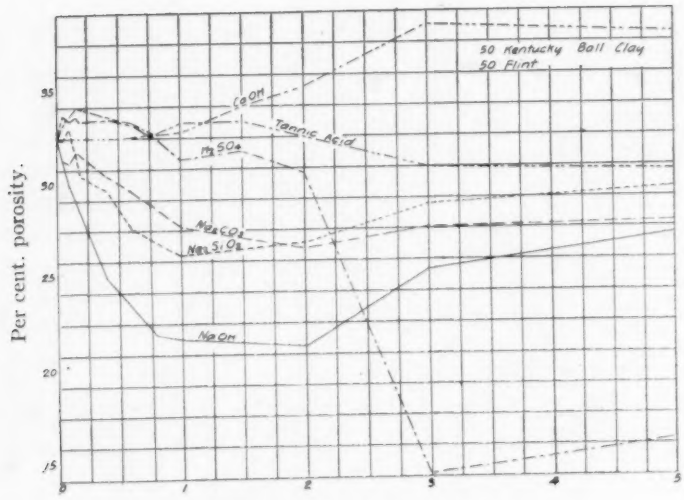
FIG. 8.—Dry Density.



Per cent. NaOH in terms of clay.

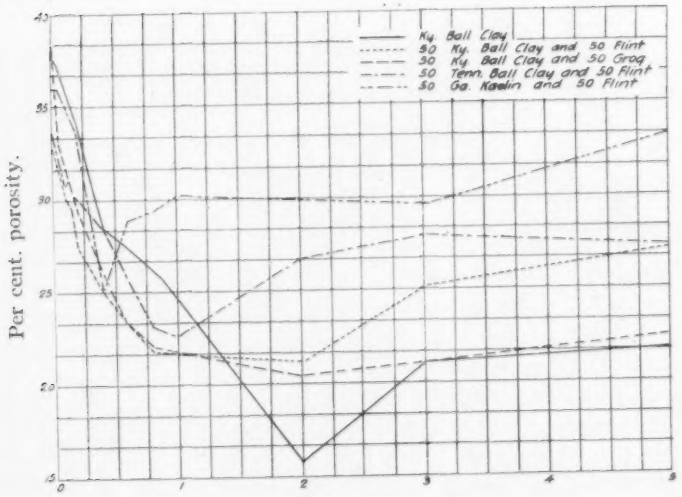
FIG. 9.—Dry Density.

Porosity.—In Figs. 10 and 11 are shown the effect of the electrolytes on the porosities of the clays in the dry condition. The porosity is decreased by the addition of the alkalies while the ad-



Per cent. electrolyte in terms of clay.

FIG. 10.—Dry Porosity.



Per cent. NaOH in terms of clay.

FIG. 11.—Dry Porosity.

dition of the acids in small percentages increases and in large percentages decreases the porosity. The porosity is increased by the addition of sodium hydroxide.

Burning Properties.—In Fig. 12 is shown the effect of sodium

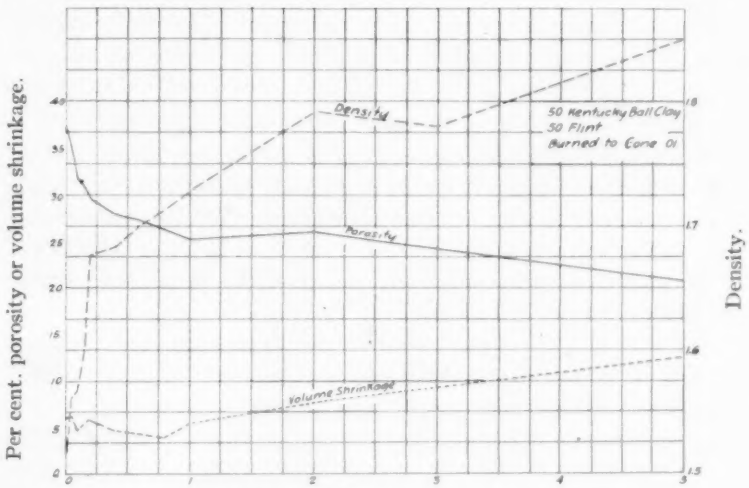
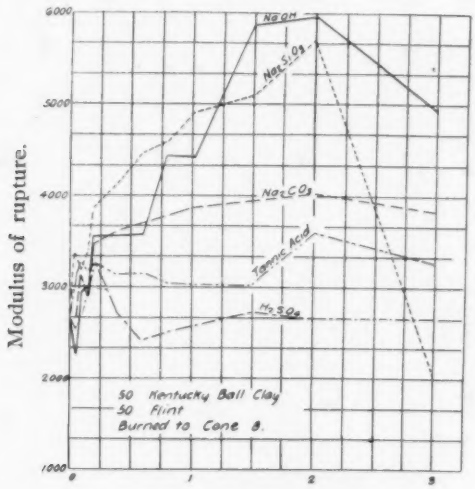


FIG. 12.—Burning Properties.

hydroxide on the burning properties of the mixture of 50 parts of Kentucky ball clay and 50 parts of flint when burned to cone 01. The volume shrinkage decreases with the addition of small quantities, up to 0.8 per cent., and is increased by the addition of larger amounts. The density is increased and the porosity decreased by the use of the sodium hydroxide. Considerable scumming and surface vitrification is noted with the addition of large percentages of sodium hydroxide.

In Fig. 13 is shown the effect of the electrolytes on the strength of the mixtures when burned to cone 8. Although the fluxing action of the alkalies increases the strength, there is a decided similarity between the burned and the raw strength data (see Fig. 1). In the burned condition, the maximum effect is pro-

duced by the alkalies when from 0.8 to 2.5 per cent. is added—the caustic soda being the most effective.



Per cent. electrolyte in terms of clay.

FIG. 13.—Burned Strength.

Summary.

The strength of clays when dry may be increased by the addition of the following electrolytes to the bodies in the plastic state, the first named being the most effective: (1) sodium hydroxide, (2) sodium silicate, (3) sodium carbonate, (4) tannic acid, and (5) calcium hydroxide. The addition of sodium hydroxide increases the strength of some of the mixtures as much as 400 per cent. Small percentages of sulphuric acid decrease the strength of Kentucky ball clay. More than 2.0 per cent. of calcium hydroxide decreases the strength of Kentucky ball clay.

The addition of from 0.80 to 2.5 per cent. of caustic soda affects adversely the molding properties of the bodies, as evidenced by their inability to retain their shape when molded, unless a very low per cent. of water is used. This is also noticeable, to a lesser extent, with additions of sodium silicate, sodium carbonate, or more than 2.0 per cent. of sulphuric acid, but is not so notice-

able with additions of calcium hydroxide or tannic acid. The addition of less than 2.0 per cent. of sulphuric acid improves the molding properties of the bodies.

In the case of the two ball clays, the addition of the alkalies lowers the per cent. water of plasticity. The addition of the acids at first increases and then decreases the water required for working the bodies. The addition of calcium hydroxide increases the water of plasticity.

The addition of from 1.0 to 2.0 per cent. of alkalies decreases the volume shrinkages of the 50 parts clay and 50 part flint mixtures with the exception of the one in which Georgia kaolin is used, in this case, the shrinkage being increased. The addition of sodium hydroxide to Kentucky ball clay increases its shrinkage. Sulphuric acid, in small percentages, increases the shrinkage and in larger percentages decreases the shrinkage. Tannic acid and calcium hydroxide increase the shrinkage.

The addition of alkalies and acids increases the densities of the clays and mixtures when dry. In amounts up to 1.0 per cent. calcium hydroxide increases the density. The addition of larger amounts decreases the density.

The porosity is lowered by the addition of alkalies and increased by the addition of acids in small percentages. Large additions of acids are accompanied by decreases in porosity. Calcium hydroxide increases the porosity.

The burning shrinkage at cone 01 is lowered by the addition of small percentages of caustic soda and is increased by the addition of larger quantities. The addition of sodium hydroxide increases the density and decreases the porosity in the burned condition.

The addition of the following electrolytes to the bodies in the plastic condition increases the strength of pieces burned to cone 8, the most effective being named first: (1) sodium hydroxide, (2) sodium silicate, (3) sodium carbonate, and (4) tannic acid.

Discussion.

MR. WHITAKER: I would like to ask Mr. Schurecht what effect ageing would have on the strength of the bodies to which

the electrolytes have been added? Would ageing still further increase the strength?

MR. SCHURECHT: We have not determined the effect of ageing on the strength of the clays, although we have determined the effect of the ageing of clay slips to which electrolytes have been added and found that it changes the viscosity. The ageing undoubtedly has an effect upon some of the other properties of the clays.

PROF. STALEY: It would appear that one of the chief advantages in having a clay of good strength would be that we might expect less cracking during drying. Several years ago, I did some work on "The Effect of the Addition of Salts on the Drying Properties of Clays."[1] In this I noted that the addition of the alkalies invariably increased the tendency of the clays to crack in drying. Possibly the pieces of very carefully dried ware, free from cracks, would have shown high strength, as in the case of Mr. Schurecht's samples, but it does not appear to be good practice to increase the possible strength of a clay by the addition of alkalies, if these additions are going to introduce drying difficulties, which make it very difficult to obtain sound ware in ordinary factory practice.

In my work, I have found that the introduction of small amounts of acid and neutral salts decreased the tendency to cracking.

MR. SHAW: My experience in the use of electrolytes in the casting of bodies bears out that of Mr. Staley. The tendency to crack was very great. I should add, however, that the plastic clays which I used were of very low grade.

COMMUNICATED DISCUSSION.

W. W. GREENWOOD: Mr. Schurecht's paper presents material which is of interest to the chemist as well as to the ceramist. In dealing with so complex a subject as the chemistry of electrolytes, great care of manipulation is essential if the results of the work are to be of much value. In this particular piece of work the writer shows such care and has handled the necessary details

[1] *Trans. Am. Ceram. Soc.*, **17**, 697–711 (1915).

of his experiments with a thoroughness which makes one feel like accepting his observations as authentic.

It would have been helpful, however, if in this paper the author could have gone a little more into detail as to the theory of his subject or if he had speculated as to why the phenomena which he observed occur. This is of value for two reasons: first, because in most cases the experimenter is better able to correla te his work with existing information—he being more familiar with the subject than the outsider; second (and this applies to the case at hand), because by not doing this Mr. Schurecht leaves the reader in a position where he may draw wrong conclusions from the facts presented.

In his summary, he states: "The dry strength may be increased by adding the following electrolytes to the bodies in the plastic state, those which are most effective being named first: (1) sodium hydroxide, (2) sodium silicate, (3) sodium carbonate, (4) tannic acid, and (5) calcium hydroxide." There is danger in assuming from this statement that the electrolytes mentioned were directly responsible for the increased strength of the dried bodies. There is nothing in the observations presented to uphold this theory. In fact, it would seem quite probable that the electrolytes are only indirectly responsible for this action. We know that their addition deflocculated the clays and the statement is made in the paper that "under the microscope it could be seen that the clays treated with alkalies consisted of very fine grains, a characteristic of deflocculated clays, while the untreated clay is much coarser." Does it not seem reasonable that the increased strength observed was at least partially due to the fineness of the particles in the treated clays? These finer particles would tend to pack closer together and consequently there would be fewer voids than in the untreated clays. In other words, this resulting strength is of mechanical origin brought about by chemical means. If such is the case—and there is much in the paper which points in this direction—the electrolytic action is simply a means towards an end—a method if you please of obtaining fine particles of clay compacted together which in turn produce greater mechanical strength.

In another part of his summary the statement is made: "The strength after being burned to cone 8 may be increased by adding the following electrolytes to the bodies in the plastic state, those which are most effective being named first: (1) sodium hydroxide (2) sodium silicate, (3) sodium carbonate, and (4) tannic acid."

Here again there is a chance for difference of opinion as to the cause of this increased strength. No doubt it is of chemical origin, but there is nothing in the paper to show whether the reagents named produced it because they were electrolytes or because of their alkali content. Binns,[1] in discussing the action of tannins on clays, notes that alkaline salts in the tannins would become operative at the higher temperatures.

How would one of these treated clays, when dry, differ from an untreated sample? Would there not be minute particles of alkaline salts about each grain? When the clay was burned these salts might be expected to exert a fluxing action and consequently a stronger and denser body would result. In the familiar process of "salt glazing," the alkali, freed from the decomposing sodium chloride, acts on the outside of the ware and glazes it—here the alkali would be in a position to act as a fluxing agent throughout the structure of the ware. So again the electrolytes themselves would be only indirectly responsible for the results noted.

The work of this paper indicates another bit of data which it would be worth while to determine. In two cases the order of effectiveness of these electrolytes is (1) sodium hydroxide, (2) sodium silicate, (3) sodium carbonate and (4) tannic acid. It would be of interest to know whether this is due to the characteristics of the reagents themselves or to their alkali content. In the two cases cited, the order of strength as electrolytes is directly proportional to the alkali content. Sodium carbonate would, in solution, hydrolyze and show a weak alkaline reaction; sodium silicate acts as a fairly strong alkali toward litmus—depending of course upon its concentration—and sodium hydrate is the strongest of all.

It may be possible that the whole theory of the effect of electrolytes on clays is really based upon the action of alkalies upon clays.

[1] *Trans. Am. Ceram. Soc.*, **6**, 57 (1904).

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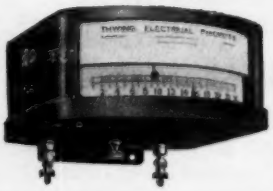

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43-43